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CONTENTS

299 **Standardization of Lubricating Oil Engine Test Methods by the Institute of Petroleum. Report of the Engine Tests of Lubricants Panel of the Institute of Petroleum**
By N. Kendall

311 **The Preparation and Infra-Red Spectra of the Three Ethyl*cyclo*-pentenes, Methylene- and Ethylidene*cyclo*pentane**
By S. Pinchas, J. Shabtai, J. Herling, and E. Gil-Av

316 **A Method for the Determination of Trace Amounts of Water in Hydrocarbons**
By A. F. Pyrah and R. S. Roberton

320 **Formula for Calculation of Lean Mixture Knocking Characteristics of Aviation Gasoline Grade 100/130**
By Raoul Niklasson

Abstracts 1310–1485

Book Reviews

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STANDARDIZATION OF LUBRICATING OIL ENGINE TEST METHODS BY THE INSTITUTE OF PETROLEUM *

REPORT OF THE ENGINE TESTS OF LUBRICANTS PANEL OF THE INSTITUTE OF PETROLEUM

By N. KENDALL † (Associate Fellow)

INTRODUCTION

WITHIN the Institute of Petroleum all matters concerning the standardization of engine test methods for evaluating lubricating oils are the responsibility of the Engine Tests of Lubricants Panel. This Panel includes representatives not only of the petroleum industry but also of other interested bodies, such as National Defence and other Government bodies, lubricating oil additive manufacturers, engine manufacturers and operators, and industrial research organizations.

The Engine Tests of Lubricants Panel is concerned only with test methods and not with product specifications, its responsibilities being:

(*a*) To develop original test methods by the voluntary collaborative effort of Panel members and others interested. This aspect will be dealt with later in the paper.

(*b*) With the agreement of the originators, to adopt, or adapt, as IP standards, any existing test methods which may be of general interest to any of our members. For example, the Caterpillar L-1 test has been adopted as an IP Standard Method, so that all relevant details of this test could be collected together and the procedure tied to a suitable rating system which enabled results from various laboratories to be compared on a common basis. Also the text of the original procedure has been altered to conform with British terminology, but technically the original test method has not been altered in any way.

(*c*) To provide assistance to anyone interested—members and others—with the various administrative and operational details involved in the running of any standard test method. For example, we acquire and disseminate all kinds of information pertinent to the operation of standard test methods; we help to overcome various kinds of operating difficulties; we investigate factors affecting repeatability and reproducibility of test methods and we try to devise ways accordingly of improving the tests in these respects; we arrange supplies of spares, lay down and distribute reference fuels and oils, organize correlation programmes, and arrange for the training of staff and operators.

The Panel has been working along these lines for about twelve years. We would now like first to outline briefly our main activities during this period and then to deal in more detail with our present position in respect of the more important and more recent items of our programme.

Outline of Activities

Shortly after the last war, when the Panel started its activities, there was a demand in Britain for a small, cheap test engine and test procedure for the evaluation of engine crankcase lubricating oils. This demand came mainly from small oil blenders, transport operators, and engine manufacturers. To meet the demand the Panel designed, and had manufactured, two types of single-cylinder test engines, one a gasoline engine, the other a diesel.

The gasoline engine, generally known as the Sunbury engine, was designed by the late R. Stansfield. Some ten years ago he took this engine to the U.S.A. and reported on it in detail to the CRC.[1] This was a four-stroke engine of 2½-inch bore × 3½-inch stroke which could be run normally aspirated or supercharged (Fig 1). Piston and crankpin temperatures could be measured whilst the engine was running, and cylinder liners of various thicknesses could be fitted which gave a means of achieving a range of temperatures of the oil film of the inner surface of the liner.

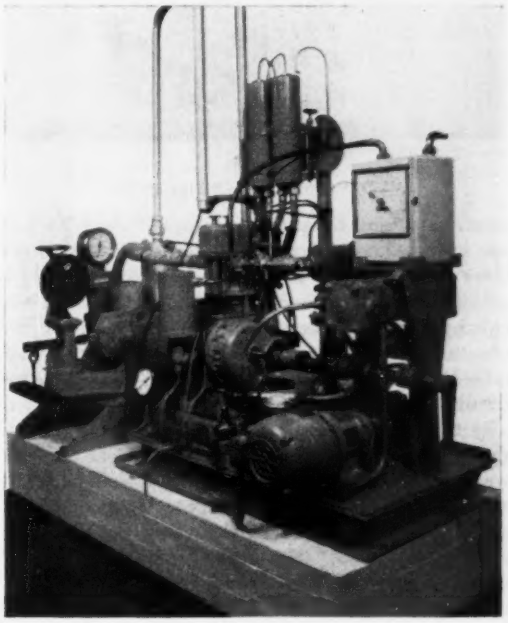

FIG 1
VIEW OF SUNBURY ENGINE

The diesel engine, known as the Shell/Ricardo

* Paper presented at SAE Summer Meeting, Atlantic City, N.J., 15 June 1959.

† "Shell" Research Ltd.

engine [2] (Fig 2), had a stroke of 6 inches and various standard top-assemblies could be fitted to the crankcase. The Gardner single-cylinder top-assembly was most commonly used.

Both the Sunbury and the Shell/Ricardo engines were designed for robustness, ease of assembly and dismantling, and accurate control of operating factors.

A number of both these engines was produced and acquired by various research laboratories, where they are still in use for research purposes. No standard lubricating oil tests were developed by the IP in either of these engines, however, because they turned out to

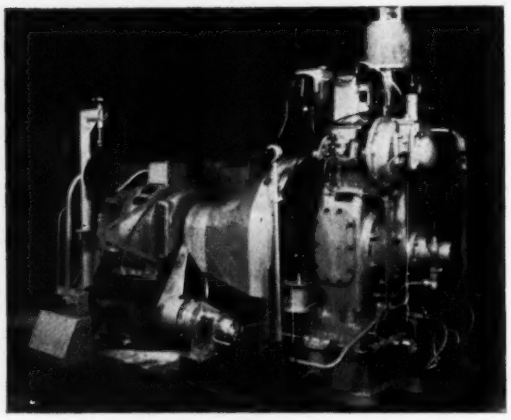

FIG 2

VIEW OF SHELL/RICARDO ENGINE

be too expensive for the very people for whom they were intended. Each engine cost something between $2000 and $3000. From this and subsequent experience we can conclude that the first cost of a test engine must not exceed about $300 in order to gain voluntary acceptance as a routine test unit in Britain and other European countries. It will be appreciated how difficult this attitude makes the task of our Panel.

By this time several member firms had developed lubricating oil screening tests in small, cheap, commercially available engines and although technically such engines were not so satisfactory as specially designed test engines, nevertheless they possessed the merit of cheapness. The Panel therefore decided to investigate some of these test methods and, if necessary, to develop them further to suit the needs of certain interests. As a first step it was decided to hold a symposium to discuss all available test methods.[3] As a direct result of papers presented at this symposium it was agreed that work to develop test procedures should be concentrated on the following three engines and associated test methods, as these appeared to be the most promising:

Gardner 1.L2 *Engine:*

A single-cylinder four-stroke compression-ignition engine. Adapted for conducting a 130-hour detergency test.

Petter AV.1 Engine:

A single-cylinder four-stroke compression-ignition engine. Adapted for conducting a 120-hour detergency test.

Petter W.1 Engine:

A single-cylinder four-stroke spark-ignition engine. Adapted for conducting a 36-hour test to evaluate copper–lead bearing corrosion.

The test methods already existing on these three engines were regarded as being basic only, and it was considered necessary to develop or improve each of them and to carry out collaborative test programmes to demonstrate the repeatability and reproducibility of the tests, to improve the reliability and suitability of the test engines, and to give some indication of the practical significance of the test results.

To facilitate this work, two Working Groups were formed, one dealing with both the Gardner and the Petter AV.1 tests, and the other dealing with the Petter W.1 test.

The task of these two Working Groups has now, after about five years, been completed and the test procedures as laid down by the Working Groups have been adopted as IP tentative standard test methods. These will be included in a separate book of Standard Lubricating Oil Engine Test Methods which is to be published by the IP.

So far as the future is concerned our Panel has no immediate plans for developing new engine test methods of our own. We would restart such activity only when some specific demand should arise. However, we are at the moment very closely associated with the development work on the CRC L-38 procedure.

Apart from all this activity of test development, our Panel has been very active in other ways, particularly in carrying out collaborative programmes to determine the repeatability and reproducibility of standard test methods and also to establish the correlation between various test methods. As a result of such work we have been able to improve the repeatability and reproducibility of some tests. Our work on the correlation between the L-1 test and the Petter AV.1 test which is summarized in this report will probably be of particular interest. A full report on the subject will, however, be given in the fairly near future, probably in the *Journal of the Institute of Petroleum*.

DEVELOPMENT OF THE GARDNER HD.4 TEST

The Gardner type 1.L2 single-cylinder diesel engine was adopted by the Shell Thornton Research Centre for the screening of diesel engine oils.

This engine (Fig 3) is specially manufactured by Norris, Henty & Gardner Ltd, Patricroft, England, as a laboratory unit at an approximate cost of $550. It is readily available and can be supplied direct coupled

to a suitable hydraulic dynamometer or electric generator for loading purposes. The engine is virtually a single-cylinder version of the manufacturer's standard LW range of engines, and thus uses standard production components, supplies of which are freely available throughout most of the world. The unit is of very robust construction and gives an extended life without the need for major overhaul. As a result of the maker's policy of ensuring interchangeability of all newly designed or developed components with those of previous design it will be very many years before this engine becomes obsolete.

An open-type combustion chamber in the piston crown gives good combustion efficiency, and the engine can thus be taken as fully representative of current British diesel engine practice. Test results

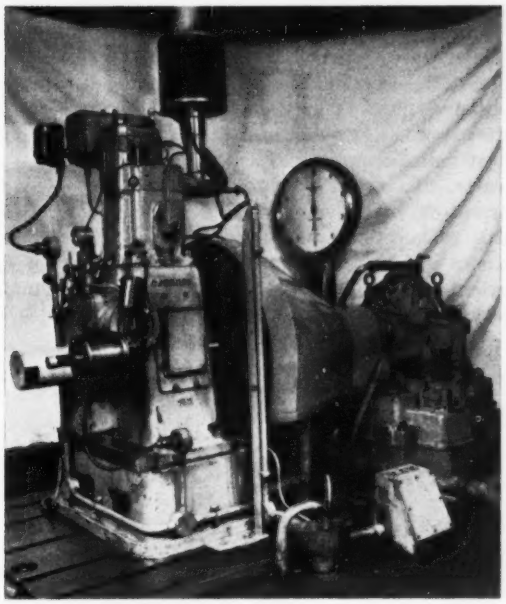

FIG 3
VIEW OF GARDNER ENGINE

can therefore be safely related to service experience in the field. The oil addition procedure used with this HD.4 test method has been specially developed to render the results obtained fully independent of the engine's oil consumption rate. At the same time, however, it has set a maximum oil consumption limit of 0·1 lb/hr, to enable the test to be fully completed. It is considered that no undue difficulty is experienced in maintaining oil consumption rates below this limit, which represents approximately 1·6 per cent of the fuel consumption rate under the test conditions.

Engine flushing run and test conditions are detailed below; the fuel used is of approximately 1 per cent sulphur content as specified for the supercharged Caterpillar 1-D test procedure:

Flushing Run: 30 minutes. (Using a previously run-in piston assembly and cylinder liner.)

Duration min	Bhp	Speed, rev/min	
5	Nil	750	During this run-in gradually bring up coolant temperature to 80° C
5	5	1000	
5	7·5	1000	
5	10	1250	
5	12·5	1500	
5	15	1500	

Note: When the engine is fitted with a new piston assembly and/or cylinder liner a six-hour break-in run is carried out with straight oil and the parts cleaned prior to running-in as above.

Test Conditions: 130 hours.

Speed	1500 rev/min
Fuel consumption	6·20 ± 0·15 lb/hr (this should give approx 16 bhp power output)
Coolant outlet temperature	80 ± 1° C—Inlet/outlet differential should not exceed 10° C
Sump oil temperature	Uncontrolled but usually between 65° and 75° C
Oil pressure	20 psi approx
Oil changes	None during test
Sump oil fill	7 lb

Note: Oil consumption throughout the test is determined at twelve-hourly intervals, after the first five hours (*i.e.* at 5, 17, 29, 41, 53, 65, 77, 89, 101, 113, and 125 hours) by stopping the engine, draining (5 min), and weighing the crankcase contents. The charge is then returned to the crankcase and fresh oil make-up equivalent to *one-half* the oil consumed during the preceding running period is added.

Before each test the engine is thoroughly cleaned according to a rigorous procedure detailed in the test method. Unless so desired, it is not necessary to fit new piston components for each test, thus reducing the total running cost. After each test the engine is drained, stripped, and rated according to the procedure fully detailed in the test method. Evaluation of the performance of the test oil is based primarily on the piston cleanliness, but wear of the piston rings and cylinder liner are recorded. Observations of the extent of oil scraper ring hole blocking and of ring sticking are made, but are not included in the rating system. The greatest sensitivity of the engine with this test procedure is obtained with oils of DEF 2101 and MIL-L-2104 quality level, thus rendering it particularly suitable for comparing the performance of oils of this general quality. Care should be exercised in assessing the quality of oils whose performances are higher or lower than this level.

So far the use of this engine has been confined to a few laboratories in the U.K., and it is thus difficult to indicate the precision of this test procedure. It has, however, been closely studied by the relevant IP Working Group and a final IP standard method of test has now been agreed for publication. This method fully details the preparation of the engine before test, the conduct of the test, and the method of examina-

tion and rating of components after test. A description of the complete engine and its preferred method of installation and instrumentation, etc., will also be included.

European operators of the Gardner engine have co-operated in all correlation test programmes organized by the IP. Details of the results obtained on this, as on other test engines, will be included in published data to be issued in the near future. However, a summary of the results obtained using the Gardner engine is given in Table I.

TABLE I

Gardner 1.L2 Correlation Results

Oil reference	Testing laboratory	Engine	Merit ratings				
			Top ring grooves 100 = clean	Other grooves 100 = clean	Ring lands 100 = clean	Piston skirt 100 = clean	Overall merit rating 100 = clean
A	W	(a)	47	50	59	87	61
			14	4	25	87	33
			60	32	42	86	58
	X	(a)	37	45	54	85	55
	Y	(a)	38	17	44	96	48
			24	13	31	96	40
	Z	(a)	32	44	46	87	52
			13	25	34	91	41
B	W	(a)	55	49	42	86	58
			37	13	11	89	88
			33	22	37	85	44
	X	(a)	51	35	54	98	59
	Y	(a)	23	3	13	93	33
			18	4	22	96	35
	Z	(a)	26	43	40	85	49
			38	39	40	78	49
C	W	(a)	42	46	59	94	60
			84	56	58	97	74
			54	61	70	99	71
	X	(a)	56	48	53	98	64
	Y	(a)	61	38	26	98	54
			48	22	36	99	55
	Z	(a)	48	43	50	86	57
			44	58	55	84	60
D	W	(a)	95	88	95	100	94
			85	86	90	99	91
			93	99	96	100	97
	X	(a)	97	94	100	100	98
	Y	(a)	82	80	88	100	87
			81	81	87	100	87
	Z	(a)	91	93	91	100	97
			78	92	89	98	89
			98	90	94	100	96
Z	Y	(a)	74	75	89	100	84
			48	66	84	100	75
	Z	(a)	55	57	60	93	66

Approximate Quality Levels of Oils referred to in Tables I and II

Oil A. Approximately of 2-104B quality level.
Oil B. Of higher quality level than 2-104B, but unacceptable for MIL-L-2104 or DEF 2101.
Oil C. Borderline oil MIL-L-2104 or DEF 2101.
Oil D. Approximate Supplement 1 quality level.

DEVELOPMENT OF THE PETTER AT.4 TEST

The Petter type AV.1 single-cylinder precombustion chamber laboratory type diesel engine (Fig 4) is manufactured by Petters Ltd, Staines, England, at a cost of $300. It was selected some twelve years ago by Anglamol Ltd (now Lubrizol Great Britain Ltd) for use in their laboratories as a small-scale low-cost test engine. This was required for the quality control performance testing of crankcase oil additive batches and for the preliminary evaluation of oil/additive blends prior to their being tested on the larger-scale Caterpillar L-1 test engine to obtain official approvals to the DEF 2101 and MIL-L-2104 military specifications.

The basic engine is in volume production and is sold widely throughout the world with all spare parts freely available. The use of critical components (such as piston, rings, and liner) manufactured to precision limits ensures consistency of test conditions with respect to oil consumption, combustion efficiency, engine component temperatures, etc. These parts are obtained separately from specified specialist manufacturers under special part numbers.

In developing a suitable test procedure (at present designated as the AT.4 Method) emphasis was laid upon the desirability of obtaining consistent and significant results in a reasonably short running period. Attempts were therefore made to increase the piston temperature while avoiding undue stress on other components which would result from running at excessive power output. The desired result was achieved by the use of kerosine as a coolant, making use of its poor heat transfer properties as compared with those of water. To avoid operating difficulties that might arise from excessive cylinder head temperatures with this coolant, a reversed flow system was adopted where coolant entered the engine at the top of the cylinder head and left the lower part of the cylinder jacket. Inlet and outlet temperatures of 78° and 85° C

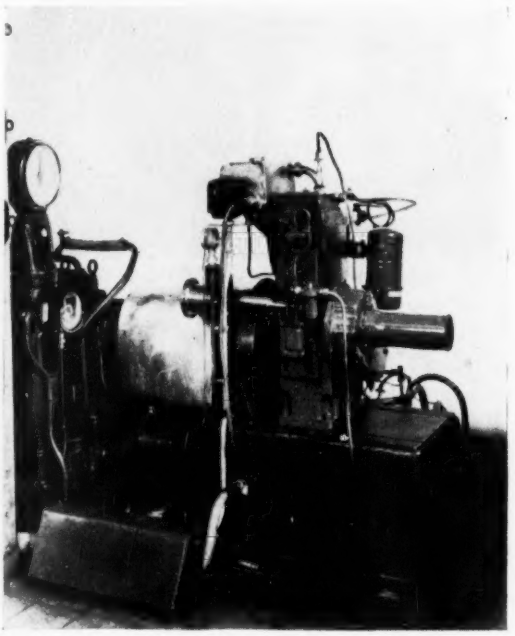

FIG 4 (*a*)
VIEW OF PETTER AV.1 ENGINE

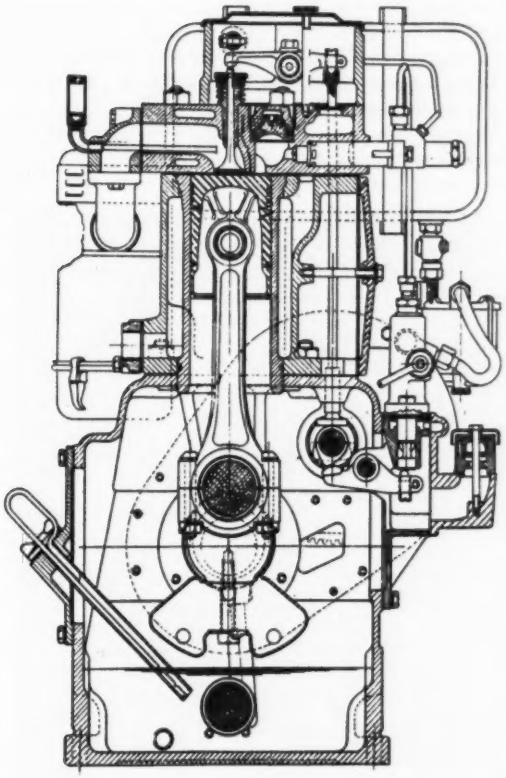

FIG 4 (*b*)
CROSS-SECTION OF PETTER AV.1 ENGINE

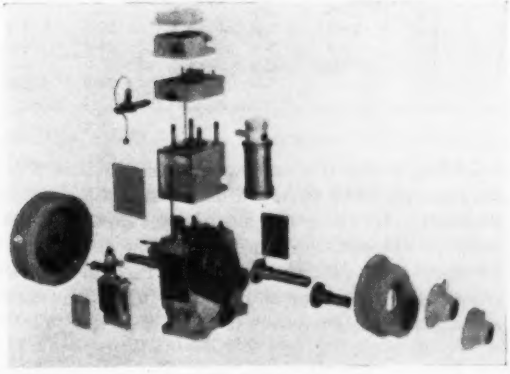

FIG 4 (*c*)
EXPLODED VIEW OF PETTER AV.1 ENGINE

respectively have been adopted, together with a sump oil temperature of 55° C. The use of these temperatures in conjunction with an engine power output of approximately 5 bhp at 1500 rev/min enables satisfactory results to be obtained with a test duration of 120 hours following an 8-hour run-in.

Pertinent details of the test and run-in procedure are given below; the same fuels are used as for the Caterpillar L-1 test engine:

Run-in—8 *hours*

Duration hr	Bhp	Inlet outlet temperature, ° C	
2	1·5	50	1500 rev/min
2	3·0	60	55° C max sump
3	4·0	75	oil temperature
1	4·9	75–80	

Test Conditions—120 *hours*

Speed	1500 ± 10 rev/min
Fuel consumption	2·4 ± 0·05 lb/hr (this should give approx 5 bhp power output)
Kerosine jacket outlet:	
Temperature	85 ± 2° C } Differential should
Inlet temperature	78 ± 2° C } not exceed 7° C
Sump oil temperature	55 ± 5° C
Oil pressure	35 ± 5 psi
Fuel spill timing	20 ± 1 degrees b.t.d.c.
Oil changes	None during test
Sump oil fill	7 lb

Before each test the engine is carefully reconditioned and fitted with a new piston and ring assembly; other components are replaced as necessary. General experience has indicated that the engine should be fully stripped, inspected, and rebuilt after each 1000 hours running. Piston rings are carefully lapped before test to obtain the specified side clearances and the plain faced top compression ring is also lapped on its periphery. The taper faced second and third compression rings are used with side lapping only. Fuel injection nozzles and pumps are serviced as appropriate.

After test the engine is drained, stripped, and rated according to a system fully detailed in the test procedure. Particular emphasis is placed upon the freedom from piston ring sticking and on the cleanliness of the piston lands and skirt after being washed free of sludge. A note is also taken of the general engine condition with respect to sludge deposits, etc., in the valve push rod and rocker arm chambers, under the piston, and on the oil pump inlet screen.

It must be emphasized that no attempt should be made to relate the ratings of individual items in the Petter AV.1 engine to the condition of the same items in the Caterpillar single-cylinder test engine. The overall conditions of the Petter engine when operated to the AT.4 procedure can, however, be used to give a good indication of an oil's performance in the Caterpillar engine as assessed for approval to the internationally recognized military and civil heavy-duty oil specifications.

Some 375 Petter AV.1 units have now been installed in laboratories located in more than fifteen countries throughout the world. As a result a vast background of experience has been built up with the engine and a good knowledge of its capabilities and limitations has been obtained. The majority of U.K. operators, to-

gether with some European operators of this engine, are represented on the relevant IP Working Group responsible for the study of this test procedure. Co-operative work over recent years has evaluated various detail points of this method, and a final agreed procedure has been prepared for publication as an IP standard method of test. A summary of results showing the repeatability, reproducibility, and sensitivity of the test is shown in Table II. Full details of this test, together with the method of examination and rating of components after test, will shortly be published; data on the precision of the results obtained will also be included. A description of the complete engine and its preferred method of installation and instrumentation, etc., will also be given.

TABLE II

Petter AV.1 Correlation Results

Test oil		A			B			C			D		
Lab	Eng	Land lacquer 10 = clean	Skirt lacquer 30 = clean	Top groove carbon 10 = clean	Land lacquer 10 = clean	Skirt lacquer 30 = clean	Top groove carbon 10 = clean	Land lacquer 10 = clean	Skirt lacquer 30 = clean	Top groove carbon 10 = clean	Land lacquer 10 = clean	Skirt lacquer 30 = clean	Top groove carbon 10 = clean
1	(*a*)	1·4	26·1	1·2	3·7	27·9	0	3·7	28·2	5·0	7·8	29·7	4·0
	(*b*)	0·8	27·2	0·7	3·3	27·9	0·7	3·8	28·0	5·2	7·8	29·8	7·0
2	(*a*)	0·4	23·5	0	1·0	26·7	4·1	1·7	27·3	3·0	5·4	30·0	6·2
	(*b*)	0·8	24·9	5·5	0·5	26·4	2·5	1·0	28·5	0	3·3	29·8	0
3	(*a*)	3·1	26·9	5·7	2·0	26·5	6·0	2·3	27·7	3·1	5·8	29·2	7·5
	(*a*)	1·3	27·0	5·4	3·5	25·6	4·7	2·7	28·0	4·0	5·0	29·7	8·5
	(*a*)	—	—	—	—	—	—	3·5	27·7	4·2	2·2	28·8	5·0
4	(*a*)	4·1	25·8	6·3	1·3	27·3	4·5	3·9	28·8	4·2	8·7	30·0	8·5
	(*b*)	2·4	28·3	3·3	2·5	28·1	6·0	0·5	28·0	5·1	7·8	30·0	—
5	(*a*)	2·7	25·9	5·5	3·7	27·0	4·0	3·7	28·5	1·2	6·2	30·0	1·7
	(*a*)	3·2	24·4	4·5	2·8	27·8	0	4·0	27·9	2·0	6·0	30·0	1·5
6	(*a*)	2·5	25·9	3·1	4·2	27·8	—	4·8	27·9	5·0	6·3	30·0	6·2
	(*b*)	1·5	24·4	0·5	2·7	27·8	—	0·7	26·9	7·0	4·7	29·7	7·4
7	(*a*)	1·2	23·4	2·5	—	—	—	2·7	25·2	6·2	9·0	29·2	9·6
	(*b*)	2·6	25·8	5·8	—	—	—	2·2	27·9	6·6	5·7	29·9	8·0
8	(*a*)	1·2	25·7	3·7	1·8	28·1	—	2·2	28·6	2·5	5·6	29·5	4·5
	(*a*)	0·8	27·0	0	1·3	28·1	—	2·7	29·1	0	7·7	30·0	5·5
9	(*a*)	3·3	26·6	5·5	1·7	28·2	—	3·7	28·9	3·2	6·4	30·0	6·5
	(*a*)	—	—	—	—	—	—	—	—	—	7·8	30·0	9·2
10	(*a*)	3·9	24·1	4·5	3·3	25·6	1·4	3·2	26·2	3·5	9·0	29·8	8·9
11	(*a*)	1·4	25·3	4·8	3·3	26·2	3·7	2·2	28·8	3·1	6·5	29·2	7·5
12	(*a*)	4·0	27·3	3·7	1·7	26·1	3·1	2·2	28·8	3·6	8·2	30·0	8·5
	(*b*)	1·7	23·8	2·5	1·0	26·2	2·5	2·3	26·2	3·8	3·8	29·9	4·5
	(*a*)	0·6	25·3	5·0	0·5	24·5	3·0	1·5	27·4	0·8	9·3	30·0	6·5
13	(*a*)	—	—	—	—	—	—	—	—	—	9·3	30·0	9·5

Because of the popularity of this test engine in Europe, during the last year an international organization was formed to study and standardize test procedures involving the Petter AV.1 engine. This body has become known as the European Council for the Study of Oil Test Procedures on the Petter AV.1 Engine. At present nine countries are co-operating in the work of this Council and each is represented by a delegate nominated by the appropriate national organizations. The IP is represented on the Council by a member of the Engine Tests of Lubricants Panel.

It is intended that the IP method and that being developed by the European Council will be fully equivalent. This test could then be used for the drafting of official oil performance specifications. Of course, neither the IP nor the European Council concern themselves with specifications.

In response to many requests a complete test rig incorporating this engine or the Petter W.1 as an alternative has been developed in co-operation with a U.K. dynamometer manufacturer. Thus laboratories intending to install such engines can purchase a fully instrumented and equipped unit which will only need connecting to water and electricity supplies and to a suitable exhaust system to be immediately ready for use. The availability of this rig will also contribute greatly to the adoption of fully standardized rigs amongst all operators of these engines.

DEVELOPMENT OF THE PETTER W.1. BEARING CORROSION TEST

The Petter W.1. Oxidation Test is mainly intended to be a laboratory test, as an alternative to the Chevrolet L-4 test, for the determination of resistance to copper–lead bearing corrosion, and oxidation stability of the lubricating oil. The piston skirt and undercrown ratings, however, can provide a considerable amount of additional information.

The Petter W.1 Laboratory Engine (Fig 5) is a single-cylinder, four-stroke, spark-ignition engine of 82·5 mm stroke and 85 mm bore and is manufactured by Petters Ltd, Staines, England, at an approximate cost of $300. While the Petter W.1 engine is in large-scale production for industrial purposes the laboratory version has a number of modifications incorporated to

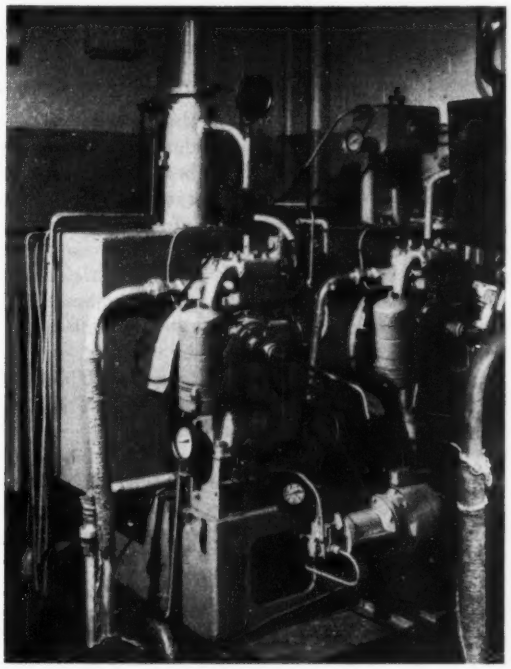

FIG 5

VIEW OF PETTER W.1 ENGINE

meet the needs of the particular test procedure adopted. These include a modified lubrication system to match that of the Chevrolet L-4 engine, Tocco-hardened crankshaft, specially made copper–lead bearings, and provision for the application of an electric sump-oil heater. However, all necessary modifications are made by the manufacturers before delivery of laboratory engines.

In order to maintain the test temperature of the lubricating oil (280° F or 137·5° C, the same as the L-4 and L-38 tests) electric heaters, made up in the form of a " frying pan " heater, are clamped to the machined underside of the crankcase, a thick metal plate being provided to prevent local hot spots developing which would otherwise cause coking of the oil. This provides the main heat, and in order to establish the necessary degree of oil temperature control a small auxiliary heater enclosed in a thick metal sheath is inserted directly into the crankcase with a control thermostat alongside. An electronic relay is used to control this heater from the thermostat.

However, in a multi-cylinder engine such as the Chevrolet L-4 the heat is generated within the engine so that to merely raise the oil temperature to that of the L-4 test does not raise the temperature of the components, in particular the bearings, to the same extent as in the L-4 engine. In order to overcome this problem a " hot spot " heater is provided and clamped to the side crankcase door. It is so arranged that the dipper attached to the connecting rod continuously throws oil over this heated surface. The temperature of this hot spot is controlled to 200° C as measured by a thermo-couple situated in the centre of the plate. This does much to increase the oxidation rate comparable to that obtained in a multi-cylinder engine test. Another important part of the test procedure which helps to control the repeatability of the test is control of crankcase ventilation, and a flapper control valve is fitted so that the crankcase operates under a small vacuum, thus helping to prevent oil leaks and also to control the amount of oxygen in the crankcase

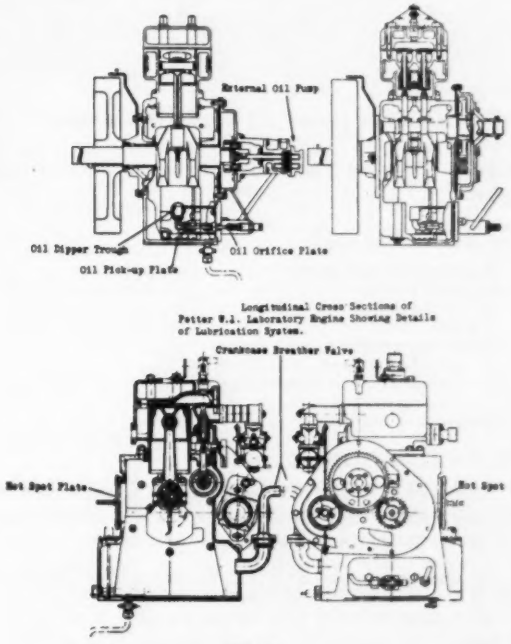

FIG 6

CROSS-SECTION OF PETTER W.1 ENGINE

atmosphere. A cross-section of the engine and details of the lubrication system are shown in Figs 6 and 7.

Development of Test Procedure

The development of the test procedure followed on from the paper given by Monsanto Chemicals Ltd at

the IP Symposium on the Engine Testing of Lubricating Oils held in 1953.[3] The test conditions and procedure as finally agreed for publication by the IP

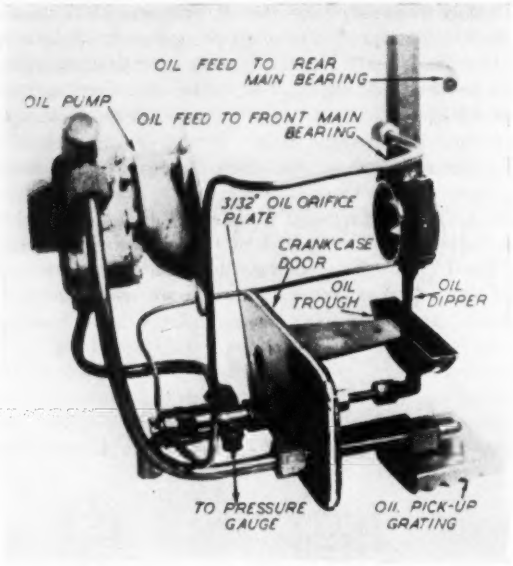

FIG 7
DETAILS OF PETTER W.1 LUBRICATING SYSTEM

Engine Tests of Lubricants Panel is summarized below.

(*a*) *Run-in Procedure*

Engine speed and power:	
First 1½ hours	1000 rev/min at 1 bhp
Next 1½ hours	1250 rev/min at 2 bhp
Final 1 hour	1500 rev/min at 3·3 bhp
Jacket temperature using water as the coolant maximum temperature in evaporative cooling system	100° C
Duration of run-in	4 hours

(*b*) *Conditions During Test Run*

The engine is started with the oil heater controls turned full on and the following test conditions are achieved as soon as possible:

Oil temperature for SAE 20, 30, 40, and 50 grades is 137·5° C ± 1° C. For SAE 10 grade 130° C ± 1° C.

Oil pressure	8 psi ± 3 psi
Speed	1500 rev/min ± 15 rev/min
Load	3·3 bhp approx
Operating time	36 hours
Coolant temperature inlet	148° C ± 2° C
Coolant temperature outlet	150° C ± 1° C
Spark advance	20° ± 1° b.t.d.c.
Oil capacity	1 kg
Oil make up	150 g oil removed and 150 g fresh oil added at 16 hours
Fuel	Currently approved IP Chevrolet L-4 gasoline
Fuel flow	45·2 sec ± 0·5 for 20 ml
Hot spot heater plate temperature	200° C ± 5° C

Co-operative Programmes Undertaken

The Petter W.1 Working Group has undertaken two main correlation programmes, the first covering four reference oils of widely differing performance levels, and the second two reference oils of MIL-L-2104A level. The first programme consisted of duplicate tests by five laboratories on four oils, these being as follows:

Reference Oil "C"	DEF 2101A oils as used in the Petter AV.1 Correlation Programme.
Reference Oil "E"	REO-7-52 CRC Reference Oil.
Reference Oil "F"	30 per cent REO-7-52 / 70 per cent REO-12-52 — CRC Reference Oil.
Reference Oil "G"	"Premium" Grade low corrosion oil.

The conclusions drawn from this programme were that while there was some considerable scatter in the numerical values obtained on bearing corrosion, in particular by some of the laboratories on the more corrosive oils, the results on oils of low corrosion were quite promising. The test had, however, fairly clearly classified the four oils into a "satisfactory" and "not satisfactory" basis, based on a limit of the order of 20 mg, bearing weight loss per whole bearing taken as the borderline pass level.

In view of the fact that the test is intended primarily for inhibited oils, which should therefore fall naturally into the "satisfactory" classification, or at least into the "borderline" category, it was decided that a further correlation programme be undertaken on two oils, both of which have been approved under the DEF 2101A Specification, and consist of formulations by two additive companies in the same base oil. These were designated Reference Oils "H" and "J."

The results of the second correlation programme showed on both reference oils that repeatability is probably of the same order as that for tests of a similar kind, such as the Chevrolet L-4 and the CRC-L-38.

A summary of the results of this second programme (Reference Oils "H" and "J") is given in Tables III and IV. These oils, which have both received DEF 2101A and MIL-L-2104A approval, are formulations in the same base oil but utilizing totally different commercial additive types.

The test procedure finally agreed is now in regular use in Europe by a number of oil companies and additive manufacturers. In the U.K. it has also been adopted in the MOS specification as an alternative to the L-4 test.

Note: Reference Oils "H" and "J" are both approved under DEF 2101A specification but are based on different commercial additives.

COMPARISON OF CATERPILLAR L-1 AND PETTER AV.1./AT.4 TESTS USING A COMMON TEST OIL

In original correlation programmes to determine the repeatability, reproducibility, and sensitivity of the Gardner 1.L2 and Petter AV.1/AT.4 test procedures, which have already been discussed, the merits of these two tests were assessed by comparison with

TABLE III

Petter W.1 Working Group—Correlation Programme
Results on Reference Oil "H"

Laboratory	A		B		C		D		E		F		G		H			J
Test number	1502	1525	2399	2405	56	58	1/14	3/14	34A	34B	—	—	—	—	1569 F2	1571 F2	1892 F2	—
Piston skirt rating	9·3	9·3	9·8	9·7	9·5	9·7	9·9	9·9	9·9	9·9	9·9	9·0	9·5	9·2	9·5	9·25	9·25	9·0
Undercrown rating	9·5	9·4	9·7	9·8	8·5	8·0	9·4	8·9	10·0	9·9	6·0	6·5	7·0	6·5	9·5	9·5	0	8·0
Oil ring rating	10·0	10·0	10·0	10·0	9·8	9·8	9·9	9·9	10·0	10·0	8·0	8·0	—	—	10·0	10·0	10·0	9·0
Copper–lead bearing weight loss, mg	4·1	5·0	5·2	3·7	9·4	6·2	14·0	15·0	5·7	5·0	19·8	29·3	16·3	17·6	54·5	52	48·5 45	51·9
Used oil viscosity increase at 100° F, %	13	20	32	33	25	23	35	37	22·1	22·1	51	51	28·2	26·3	46	42	19	39 *
Oil recovery, oz	36	36	27	32	—	—	26	24	34	36	29	30	26	28	17	24	9	—

Averages	Rating	All labs	Labs H and J
	Piston skirt	9·4	—
	Undercrown	8·6	—
	Oil ring	9·3	—
	Bearing weight loss, mg	20·2	11·2
	Viscosity increase, %	32·8	29·8

* Visc increase based on Engler at 50° C.

TABLE IV

Petter W.1 Working Group—Correlation Programme Appendix I to Minutes of Meeting No. 4 Results of Reference Oil "J"

Laboratory	A		B	C		D		E		F		G		H		
Test number	1642	1644	2577	2793	51	57	3/16	1/16	33	—	—	—	8·5	1567 1F2	1567 F2	1691 F2
Piston skirt rating	9·8	9·5	9·8	9·9	9·3	8·5	9·9	9·9	9·8	9·5	8·5	8·8	7·0	8·5	9·75	9·5
Undercrown rating	9·5	8·5	9·2	8·9	7·5	7·3	7·0	7·5	8·5	5·0	5·0	5·5	—	4·5	8·5	8·0
Oil ring rating	10·0	10·0	10·0	10·0	9·8	9·8	9·9	9·9	10·0	8·0	8·0	—		—	9·0	—
Copper–lead bearing weight loss, mg	19·0	17·0	10·7	13·1	6·0	9·0	6·0	7·0	18·6	142	37·4	10·0	2·0	14·3	4·0	3·0
Used oil viscosity increase at 100° F, %	17	22	37	51	22	23	32	32	27	20	38	21·2	20·6 25	26·5	27	31
Oil recovery, oz	32	38	—	—	—	—	24	28	32	30	29	28		23	26	24

Averages	Rating	All labs
	Piston skirt	9·4
	Undercrown	7·0
	Oil ring	9·3
	Bearing weight loss, mg	12·65 *
	Viscosity increase, %	27·5

* Excluding Lab F142 mg result.

single Caterpillar L-1 tests on each of the four reference oils used. While, as already demonstrated, the results showed that both test procedures broadly grouped the oils in the same way as the Caterpillar L-1 test, because of the very limited Caterpillar data obtained on each of the four oils it was not possible to compare the repeatability and reproducibility of these tests with those of the Caterpillar L-1 test. Neither was it possible to determine whether the tests were as discriminating as the Caterpillar L-1 test.

Therefore, immediately following the completion of the original correlation programme it was decided to carry out a further correlation programme in which as many laboratories as possible were asked to conduct Caterpillar L-1, Gardner 1.L2, and Petter AV.1/AT.4 tests on a common batch oil. Due to shortage of supplies it was not possible to use one of the previous correlation oils for the purpose. The correlation oil which was chosen, designated Correlation Oil "Z," was believed to be borderline with respect to DEF 2101B and MIL-L-2104A requirements. It was considered that an oil of this quality level was the most suitable for comparing the three test methods; moreover, as the military specifications have become world-wide quality standards for additive type oils, most laboratories require engine tests to predict oil performance in relation to the requirements of these specifications.

(a) *Caterpillar L-1 Test Results*

It is believed that it is the first time that Caterpillar engines have been included in a correlation programme

of this type, at least since the inception of the MIL-L-2104 and DEF 2101 specifications in 1950 and 1951 respectively. When it became known that the Institute of Petroleum was undertaking this programme, requests to participate by conducting Caterpillar L-1 tests on the common oil were received from several laboratories who do not normally co-operate in IP programmes. In all, 28 laboratories agreed to participate by conducting Caterpillar L-1 tests on Correlation Oil "Z." Thirteen of these were in the U.K., nine in other European countries, four in the U.S.A., one in Canada, and one in South Africa. While one or two of these laboratories were new to the engine testing of lubricating oils, the large majority were well-established laboratories employing personnel fully experienced in engine testing techniques. Many of them made more than one Caterpillar L-1 test on Correlation Oil "Z," and up to the present time 43 individual results have been obtained and a further six tests have yet to be completed.

In all the U.K. laboratories and some of the other European laboratories the fuel used was taken from a common stock laid down by the Institute of Petroleum for the use of laboratories conducting engine tests.

TABLE V

Caterpillar L-1 Results on Correlation Oil 2

Lab. no.	Engine	Top ring groove filling, %	Demerit ratings (IP 124/method)						Estimated piston condition relative to DEF and MIL requirements
			Ring groove deposits			Ring land deposits			
			2nd	3rd	4th	1st	2nd	3rd	
1	(*a*)	8·0	0	0	0	Trace	0	0	Pass
	(*b*)	25·0	0	0	0	Trace	0	0	Borderline fail
	(*a*)	1·5	0	0	0	Trace	0	0	Pass
	(*c*)	38·0	0	0	0	Trace	0	0	Fail
	(*a*)	27·0	0	0	0	Trace	0	0	Fail
2	(*a*)	1·0	Trace	0	0	Trace	Trace	Trace	Borderline pass
	(*b*)	1·4	Trace	0	0	Trace	0	0	Pass
3	(*a*)	23·0	0	0	0	0·15	0	0	Borderline fail
4	(*a*)	15·8	0·03	0	0	0	0	0	Fail
	(*b*)	36·3	0·02	0	0	0	0	0	(Piston not available)
5	(*a*)	12·0	0	0	0	0·02	0	0	Borderline pass
6	(*a*)	Test not yet completed							
7	(*a*)	12·0	0·3	0	0	0·2	0	0	Borderline pass
8	(*a*)	1·0	0·1	0·1	0	0·2	0	0	Borderline pass
9	(*a*)	2·6	0	0	0	0·02	0	0	Pass
10	(*a*)	25·0	1·2	0·10	0	0·75	0·5	0·5	(Piston not available)
11	(*a*)	11·0	0	0	0	0·2	0	0	Pass
	(*b*)	29·0	0	0	0	0·02	0	0	(Piston not available)
12	(*a*)	1·0	0·03	0·03	0	1·45	0·09	0·07	Borderline pass
13	(*a*)	7·0	0·01	0·01	0	0·16	0	0	Borderline pass
	(*b*)	14·0	0·04	0	0	0·21	0	0	Borderline fail
14	(*a*)	3·9	0	0	0	Trace	0	0	Pass
	(*b*)	2·8	0	0	0	Trace	0	0	Borderline pass
15	(*a*)	1·0	0·01	0	0	0·62	0·23	0·2	Borderline pass
	(*b*)	1·8	0·02	0	0	1·36	0	0	Borderline pass
16	(*a*)	1·5	0	0	0	0	0	0	Pass
	(*b*)	0·2	0	0	0	0	0	0	Borderline pass
	(*c*)	1·0	0	0	0	—	—	0·1	Fail
	(*d*)	2·0	0	0	0	0	0	0	Pass
	(*e*)	0	0	0	0	0	0	0	Borderline pass
17	(*a*)	20·0	0	0	0	0·2	0	0	(Piston not available)
	(*a*)	10·0	0·1	0	0	Trace	0	0	(Piston not available)
18	(*a*)	6·5	0·02	0	0	0·16	0	Trace	Borderline pass
	(*a*)	15·0	Trace	0	0	0·1	0	0·02	Borderline pass
19	(*a*)	28·0	0·34	0	0	0	0	0	Fail
20	(*a*)	16·0	0	0	0	0	0	0	Borderline pass
21	(*a*)	4·0	0·15	0·03	0	0·2	0·01	0·01	Fail
	(*b*)	37·8	0·01	0	0	0·85	0·05	0·05	(Piston not available)
22	(*a*)	0·5	0	0	0·01	0	0	0	Borderline fail
	(*b*)	22·0	0·02	0	0	0·03	0	0	Fail
23	(*a*)	Test not yet completed							
24	(*a*)	10·0	0·1	0	0	0	0	0	Borderline pass
25	?	Test not yet completed							
	?	Test not yet completed							
	?	Test not yet completed							
26	(*a*)	23·0	Trace	0	0	Trace	0	0	Borderline fail
27	(*a*)	6·0	0	0	0	0	0	0	Pass
28	(*a*)	11·0	0·1	0	0	0·1	0	0	Borderline fail

(*a*), (*b*), (*c*), etc., denote different engines in the same laboratory.

In other laboratories the fuels used meet the requirements specified for Caterpillar L-1 test fuel.

A summary of the results so far obtained is given in Table V. In addition to numerical ratings it will be seen that the results have been assessed in terms of DEF and MIL requirements. However, it must be emphasized that the pistons were not officially assessed by the respective Panels and the gradings were assigned by actual inspection of the pistons carried out by a number of representatives who have had considerable experience in assessing results for DEF 2101B and MIL-L-2104A approvals.

(b) Petter AV.1/AT.4 Test Results

Petter AV.1/AT.4 tests on Correlation Oil " Z " were carried out in eighteen laboratories, ten of which were in the U.K. and eight in other European countries. Again many of the laboratories carried out more than one test and, in all, 33 individual tests were made. The fuel for these tests was that used by the various laboratories for the Caterpillar L-1 tests, and again the large majority used the IP reference fuel.

The results of the Petter AV.1/AT.4 tests are summarized in Table VI. Again the engine components

TABLE VI

Petter AV.1 Results on Correlation Oil " Z "

Lab code number	Test number	Centralized IP merit ratings				DEF/MIL acceptability based on	
		Crown cutting	Piston land lacquer	Piston skirt lacquer	Top groove carbon	Laboratories ratings	Centralized ratings
1	EP.101	4·7	4·2	28·9	3·5	Borderline	Pass
2	P2/52	4·9	4·7	24·9	2·1	Pass	Borderline
	P2/53	4·7	6·0	30·0	7·0	Pass	Pass
3	89-6-2	4·3	6·0	29·2	8·7	Borderline	Pass
6		Tests not yet completed					
7	P3/74	4·0	2·6	28·7	4·8	Borderline	Borderline
8	P4/69	3·5	2·0	29·5	6·3	Pass	Borderline
9	PA.81	4·3	1·7	29·6	4·3	Borderline	Borderline
10		Tests not yet completed					
11	499	4·2	2·4	29·8	7·0	Borderline	Borderline
	502	—	—	—	—	Pass	—
12	P.267B	4·3	4·5	29·7	6·8	Pass	Pass
13	1/58	4·0	1·9	29·8	5·0	Borderline	Borderline
14	3130	4·0	3·5	28·7	5·5	Borderline	Borderline
	3212	4·3	4·0	28·8	9·0	Pass	Pass
	3156	4·1	5·3	27·7	8·3	Borderline	Borderline
15	700	4·3	5·8	29·6	6·0	Pass	Pass
	701	4·4	8·1	29·3	6·5	Pass	Pass
	724	4·9	5·5	29·7	6·3:	Pass	Pass
	725	4·2	5·3	29·1	4·8	Pass	Pass
16	2094	4·3	2·2	29·8	3·0	Pass	Borderline
	2183	4·1	2·1	29·2	3·0	Borderline	Borderline
	2226	4·5	4·6	29·5	3·5	Pass	Pass
17	76	4·1	2·0	29·5	3·5	Borderline	Borderline
	77	4·5	0·6	25·8	4·5	Fail	Fail
	79	4·2	1·6	26·1	4·3	Fail	Fail
	80	4·2	2·4	29·2	3·4	Borderline	Borderline
18	2/14	4·7	5·0	28·9	6·3	Borderline	Pass
	2/15	4·3	1·8	29·0	6·5	Borderline	Borderline
	3/30	4·2	5·8	29·3	5·5	Borderline	Pass
19	—	—	—	—	—	Pass	—
20		Tests not yet completed					
22	1–115	3·3	2·1	29·3	3·8	Borderline	Borderline
	2–135	4·2	3·8	29·4	5·5	Borderline	Borderline

were examined by a small group of experts and graded in relation to the previously established correlation between Petter AV.1/AT.4 results and DEF and MIL requirements.

(c) Gardner 1.L2 Test Results

Insufficient results have as yet been obtained on Correlation Oil " Z " in this engine to enable the comparison to be made with those from Caterpillar L-1 and Petter AV.1/AT.4 tests.

(d) Comparison of Results

When all laboratories have completed the tests on Correlation Oil " Z " it is intended to set up a small committee to review and analyse the results, together with all available data concerning engine measurements taken before and after tests, operating conditions, and readings taken during test, to determine whether any reasons can be found for the spread in results shown by both the Caterpillar and Petter engines.

It is of some concern to our Panel that considerable significance appears to be placed on differences between Caterpillar L-1 results, which have now been shown to be well within the repeatability and reproducibility limits of the test. It is appreciated that no engine test can be made as precise as most chemical and physical tests carried out in the analytical laboratory, but it is believed that by careful study of the data obtained in this programme it will be possible to suggest ways in which the actual reproducibility of the L-1 test may be brought into line with the presently reputed reproducibility. At least that is the hope of our Panel, and the ultimate goal is to be able to assign to the test practical reproducibility and repeatability limits.

The Petter AV.1 test, on the other hand, has always been considered a secondary engine test to the Caterpillar from the standpoint of precision. It is a small engine with inevitably somewhat wider manufacturing tolerances when these are considered as a percentage of the total clearances, etc., and it is a short test which would be expected to accentuate small differences between components and operating conditions. The idea behind the development of the Petter AV.1/AT.4 test had been to provide means of sorting oils into broadly the same quality levels as shown by the Caterpillar L-1 test, but nevertheless it is possible that the forthcoming review of the Petter results, engine variables, and operating conditions may lead to better repeatability and reproducibility than those shown by the present results.

However, comparing the estimated ratings with respect to DEF and MIL requirements for the two engine tests, out of the 37 Caterpillar pistons examined, nine were considered to Pass, 21 to be Borderline, and seven to Fail. Of the 31 Petter pistons reviewed 13 were considered to Pass, 16 to be Borderline, and two to Fail. From this comparison it appears that the repeatability and reproducibility of the Petter AV.1/AT.4 are of the same order as for the Caterpillar L-1 test.

CO-OPERATION IN CRC PROGRAMMES TO DEVELOP A TEST IN THE CLR ENGINE TO REPLACE THE L-4 TEST

Our Panel has been interested in the development of the CLR oil test engine ever since its inception. In 1955, at a meeting in London, the CRC described the design details of the engine, reported progress, and explained the aims of the project.[4] By 1959 a number of laboratories in the U.K. and other European countries who participate in the activities of the IP Engine Tests of Lubricants Panel expressed interest in the CLR L-38 technique. In view of this an IP/CLR Working Group was set up and contact was made with the CRC informing this organization of our interest in their work and the readiness of laboratories in Europe to take part in the development of a satisfactory test. As a result the IP/CLR Working Group has been co-opted as the European Area for the participation in the CRC/CLR programmes, and the Chairman of this Group nominated as the European Area Co-ordinator.

Up to the present time only two or three laboratories have actually been able to provide test data. Several other laboratories have had considerable difficulties in setting up their engines and in obtaining current spare parts; but for these difficulties, considerably more data would have been submitted.

However, it has already been possible for various European laboratories to submit suggestions and comments to the CRC Motor Oil Oxidation Tests Group through the Chairman of the IP/CLR Group, and it is hoped that in the very near future up to eight laboratories in Europe will be fully active in the work.

CONCLUSION

In this report we have attempted to give a brief factual account of the aims and work of our Panel since its inception and to describe the current status of our progress on the standardization of lubricating oil engine test methods. Before concluding it should pointed out that anything that is done in the U.S.A. in the field of standardizing and generally adopting lubricating oil engine test methods is of vital interest and concern, not only to Britain but also to other European countries. Any lubricating oil specification which becomes of commercial interest in the U.S.A. automatically, as a result of the nature of the international structure of the petroleum market, has a considerable impact on Europe and indeed upon most of the rest of the world. It is for this reason that for some time we have been seeking closer contact and greater collaboration with those in the U.S.A. engaged in activities corresponding to our own. From our contacts with similar bodies in other European countries and through our participation in the European Council we have found that international collaboration is very active and increasing. We therefore feel we have much to offer, as well as to gain, by achieving greater collaboration with the U.S.A. and that this will contribute materially towards the aims of world standardization and hence to the common good of both the petroleum industry and petroleum users. European participation in the CRC L-38 test development programme has been a good start in this direction and has given us a good idea of what we could, and should, do in the future. It is highly desirable that everything possible should be done to further understanding, contact, and collaboration between the U.S.A. and Europe on all matters concerning the development and standardization of lubricating oil engine test methods.

In conclusion, I would like to acknowledge the very considerable amount of work done by members of our Panel and its Working Groups, and the following, who have not only contributed so much to our achievements in the past but have also participated with me in the preparation of this report:

C. O. R. Bell (Monsanto), Chairman of the Working Group for the development of the Petter W.1 Engine Test.

J. C. Cree (British Petroleum), Secretary of the Panel, Chairman of the Institute of Petroleum L-38 Working Group, and Area Co-ordinator for Europe in the CRC programme for the development of the L-38 engine.

A. Towle (Lubrizol), Chairman of the Working Group for the development of the Petter and Gardner 1.L2 engine tests, and also Chairman of the Engine Operators' Working Group for the study of the instrumentation and equipment associated with lubricating oil engine tests.

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THE PREPARATION AND INFRA-RED SPECTRA OF THE THREE ETHYL*CYCLO*PENTENES, METHYLENE- AND ETHYLIDENE*CYCLO*PENTANE *

By S. PINCHAS,† J. SHABTAI,† J. HERLING,† and E. GIL-AV †

SUMMARY

Pure samples of the three isomeric ethyl*cyclo*pentenes, methylene- and ethylidene*cyclo*pentane were prepared. The synthesis of 4-ethyl*cyclo*pentene is reported for the first time. The infra-red spectra of all the compounds are reproduced and the absorptions of the C—H and C=C groups are discussed. A salient feature is the presence of a well-developed doublet in the 1600–1700-cm^{-1} region of 3-ethyl*cyclo*pentene.

DATA available in the chemical literature on cyclic and semi-cyclic olefins are incomplete and sometimes contradictory. A number of the simplest members of this class have not been prepared in a state of high purity, and the synthesis of others, *e.g.* 4-ethyl*cyclo*pentene, has not yet been recorded.

The present paper is concerned with the preparation and infra-red absorption of the three isomeric ethyl*cyclo*pentenes, methylene- and ethylidene*cyclo*pentane.

The compounds studied were obtained from the corresponding alcohols or acetates, with the exception of 3-ethyl*cyclo*pentene, which was prepared by condensation of ethylmagnesium bromide with 3-bromo*cyclo*pentene. In most cases the products consisted of mixtures of olefins from which the pure compounds were separated by fractional distillation through a spinning band column or, where this method was inadequate, by gas–liquid chromatography over a saturated silver nitrate/glycol solution [1, 2] (4-ethyl*cyclo*pentene and methylene*cyclo*pentane). Gas–liquid chromatography was also used to determine the purity of the products. Samples of more than 99 per cent purity were obtained for all compounds, except 4-ethyl*cyclo*pentene (98·5 per cent).

The structure of 1-ethyl*cyclo*pentene, 3-ethyl*cyclo*pentene, methylene- and ethylidene*cyclo*pentane followed from their method of preparation and the agreement of their physical properties with available literature data for carefully purified compounds.[3–5] The position of the double bond in ethylidene*cyclo*pentane was confirmed, in addition, by ozonolysis. The structure of 4-ethyl*cyclo*pentene was proved by oxidation to β-ethylglutaric acid.

In analogy with the corresponding methyl isomers,[1, 6] it was found that the boiling point of 4-ethyl*cyclo*pentene (98·2°) was practically identical with that of the 3-ethyl isomer (98·1°). This is in disagreement with literature data,[7] based on unpublished work,[8] according to which the boiling point of 4-ethyl*cyclo*pentene (" 106° ") is nearly identical with that of 1-ethyl*cyclo*pentene (106·5°). It seems likely that, as in the case of the methyl isomers,[6] confusion has arisen as to the identity of the 4-ethyl isomer, because the latter had apparently isomerized to the more stable 1-ethyl*cyclo*pentene under the conditions of its expected formation.

TABLE I

Frequencies of Infra-red Absorption Maxima (cm^{-1})

1-Ethyl-*cyclo*-pentene	3-Ethyl-*cyclo*-pentene	4-Ethyl-*cyclo*-pentene	Ethylidene-*cyclo*-pentane	Methylene-*cyclo*-pentane
686	714	688	698	764
754	718	766	730	876
773	752	781	752	882
821	777	809	773	922
858	788	835	805	979
888	847	893	838	1014
904	910	927	880	1132
944	935	970	897	1149
975	981	1020	944	1215
1026	1014	1055	971	1285
1052	1064	1088	1020	1432
1076	1094	1216	1066	1651
1099	1171	1262	1128	1755
1132	1210	1317	1178	2620
1205	1261	1351	1213	2885
1274	1285	1379	1227	2935
1294	1316	1460	1282	3070
1311	1361	1561	1306	
1375	1377	1616	1378	
1460	1458	1671	1439	
1651	1616	2850	1550	
1910	1650	2910	1603	
2485	2870	3065	1636	
2880	2920		1682	
3040	3060		1829	
	3415		1869	
			2550	
			2605	
			2890	
			3490	

The results of the infra-red absorption measurements are given in Table I and Figs 1–5.

* MS received 9 March 1959.

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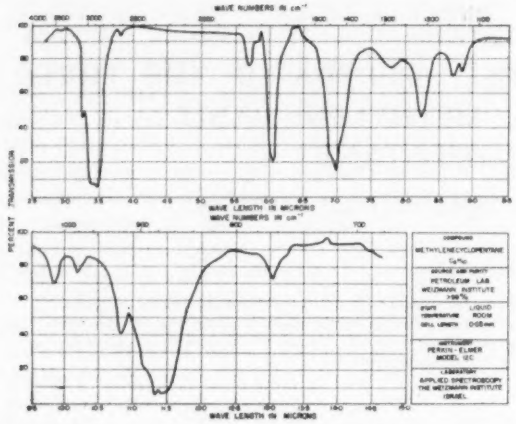


FIG 1

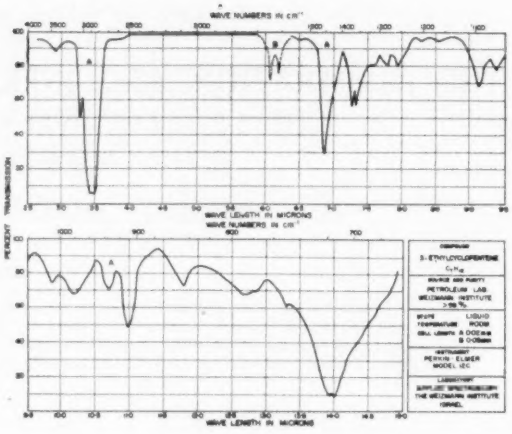


FIG 4

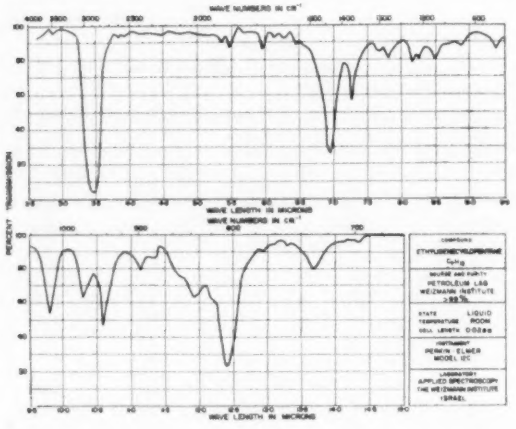


FIG 2

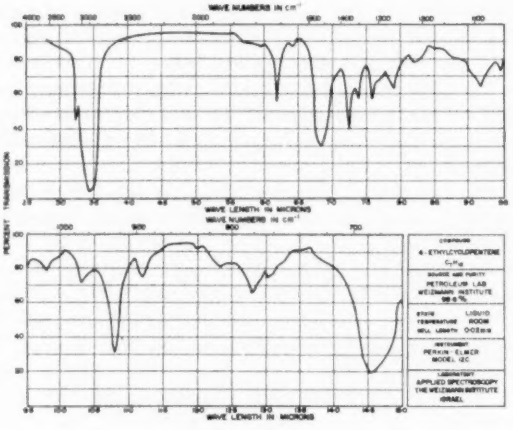


FIG 5

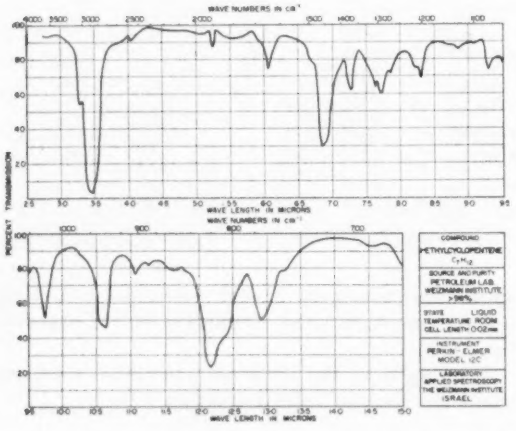
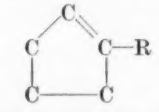


FIG 3

The =C—H *Absorption*

In agreement with the reported (HC=)C—H stretching band of *cyclo*pentene (3065 cm^{-1}),[9] the absorption bands due to the stretching of this group in 3- and 4-ethyl*cyclo*pentene were found at 3060 and 3065 cm^{-1}, respectively. These values are a little higher than that of the aliphatic (HC=)C—H group (about 3030 cm^{-1}),[10] because of the strain of the *cyclo*pentene ring, which tends to *increase* the strength of *exocyclic* bonds,[11] such as C—R in (I):



. . . . . (I)

If one of the hydrogen atoms of the group HC=CH is replaced by an alkyl substituent, the stretching frequency of the =C—H unit is decreased to about 3040 cm^{-1} (1-ethyl*cyclo*pentene, 3040 cm^{-1}; 1-methyl-*cyclo*pentene,[12] 3040 cm^{-1}). In the series of aliphatic

olefins [13] an alkyl substituent decreases this frequency to 3019 cm^{-1}. The role of the alkyl substituent seems to consist in polarizing somewhat the double bond, thereby making the =C—H unit more like a saturated —C—H group, the stretching frequency of which is much lower (~2900 cm^{-1}).

It is interesting to note that ethylene*cyclo*pentane does not show, with the resolution of a NaCl prism, a separate =C—H absorption band. It would appear that, due to the lower frequency of the non-*cyclo*-

pentenic H—C=(C) unit and the relative weakness of a single =C—H absorption in the presence of 11 saturated —C—H bonds, its expected band is totally merged with that of the saturated —C—H groups at about 2900 cm^{-1}.

The HC=CH *Absorption*

The HC=CH stretching frequency of the 3- and 4-ethyl*cyclo*pentene at 1616 cm^{-1} is in agreement with the value of 1617 cm^{-1}, reported for the Raman spectrum of the 3-isomer,[14] and the 1612 cm^{-1} band of *cyclo*pentene itself. The low value of this frequency is again due to the strain of the ring, which in the case of *endocyclic* bonds *lowers* the stretching frequencies.[11]

In 1-ethyl*cyclo*pentene this band appears at 1651 cm^{-1},* in agreement with a corresponding Raman frequency [14] of 1653 cm^{-1}. The value of 1651 cm^{-1} was also found for the HC=CH absorption of 1-methyl*cyclo*pentene.[12] This shift of the HC=CH frequency brought about by alkyl substitution is in accord with the observations made in the series of the aliphatic olefins (see Table II). The effect seems to be connected with the big difference in the stretching frequencies of the C—H and C=C bonds. The C—H bond has a frequency sufficiently removed from that of the C=C stretching vibration as to behave towards it practically like a rigid link. Hence the mass of each of the two olefinic vibrating units is not that of —C— (12) but nearer to that of —CH (13). On the other hand, the C—C bond, the stretching frequency of which is lower,[18] *i.e.* between 800 and 1150 cm^{-1}, does change its length during the vibration and, therefore, in the case of monoalkyl substitution, the mass of one of the olefinic vibrating units is very nearly that of —C— (12). As a result the R—C=CH frequency is higher than that of the HC=CH group.

Methylene*cyclo*pentane absorbs at 1651 cm^{-1}, *i.e.* at approximately the same frequency as the corresponding aliphatic olefin R′R″C=CH_2. The value for this frequency is in agreement with the Raman band,[14] found at 1653 cm^{-1}, and seems to be more correct than that given by Lord and Miller [19] (1657 cm^{-1}).

Ethylidene*cyclo*pentane, because of the effect of the alkyl substitution, discussed above, shows its absorption frequency at 1682 cm^{-1}, which is somewhat higher than the expected value (1670 cm^{-1}) for the aliphatic analogue (see Table II).

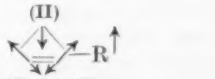
TABLE II

The C=C *Stretching Frequency in Different Groups* [17]

Olefin type	Frequency (cm^{-1})
$CHR=CH_2$	1643
$CR'R''=CH_2$	1653
cis $CHR'=CHR''$	1657
$CR'R''=CHR'''$	1670

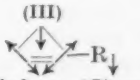
Apart from the C=C band at 1616 cm^{-1}, the 3-ethyl*cyclo*pentene shows a second, somewhat stronger band, at about 1650 cm^{-1}. 1-Ethyl*cyclo*pentene has, however, only one medium and one very weak absorption in this region. A well developed doublet, at the same places, appears also in the spectrum of 3-methyl*cyclo*pentene.[6] *cyclo*Pentene itself [9] shows only a single band in this region. It seems, therefore, that this doublet is characteristic for the presence of a 3-alkyl substituent in the *cyclo*pentene ring and can be used for its identification. The 1616- and 1650-cm^{-1} bands belong probably to the alkyl-in-phase and alkyl-out-of-phase C=C stretching modes of vibration, respectively (II, III):

(II)



Alkyl-in-Phase

(III)



Alkyl-out-of-Phase

The 1612 cm^{-1} frequency of *cyclo*pentene itself, which corresponds to these bands, was assigned to the similar ω_5 (IV) mode of vibration by Reitz [20] and Lecomte and co-workers: [15]

(IV)

ω_5

The very weak band of 4-ethyl*cyclo*pentene at 1671 cm^{-1} may be due to an analogous alkyl-out-of-phase vibration, being, however, much weaker because of the higher symmetry of this isomer.

If this assignment is correct, such a splitting of the C=C frequency should also be observed for *cyclo*hexenes. The reported spectrum of 3-methyl*cyclo*hexene [21] shows, indeed, such a doublet at 1648 and 1682 cm^{-1}. The frequencies are higher than in the *cyclo*pentenes, because of the lack of strain in this case, but the difference between the two bands is the same

* Cauquil, Delay, and Lecomte [15] have reported different values from ours for the infra-red bands of 1-ethyl*cyclo*pentene, but found it difficult to reconcile them with the Raman lines, measured on a different sample many years previously.[16] In particular, they found a band at 1730 cm^{-1}, but none at 1651 cm^{-1}. The preparation of the sample, the infra-red spectrum of which was measured, is not reported. Apparently, it was impure, the absorption at 1730 cm^{-1} being possibly due to the presence of *cyclo*pentanone.

(34 cm^{-1}) for both 3-ethyl*cyclo*pentene and 3-methyl-*cyclo*hexene. In the spectrum of the relatively more (but not fully) symmetrical 4-methyl*cyclo*hexene,[22] there also appears a doublet at 1653 and 1687 cm^{-1}, the latter band being, however, much weaker than the corresponding absorption of the 3-methyl isomer.

Other Absorptions

The strong and characteristic bands of 3- and 4-ethyl*cyclo*pentene at about 714 and 688 cm^{-1}, respectively, should also be mentioned, since they can, no doubt, be used for identification and quantitative determination of the isomers. They belong, of course, to the *cis* H—C (=CH) out of plane bending vibration.[10]

EXPERIMENTAL

*Methylene*cyclo*pentane*

*cyclo*Pentyl carbinol was prepared by the action of formaldehyde on *cyclo*pentyl magnesium bromide, according to Vogel.[23] The carbinol was esterified with acetic anhydride and the resulting acetate pyrolysed at 570° and 15 sec contact time in a Pyrex glass tube of 25 mm diameter and 25 cm length, filled with Pyrex beads. The olefinic product contained 70 per cent methylene*cyclo*pentane and 30 per cent 1-methyl-*cyclo*pentene. About 0·25 g of methylene*cyclo*pentane of 99 per cent purity was prepared by gas–liquid chromatography of the mixture on a column of 2 m length and 10 mm internal diameter, filled with a saturated silver nitrate/glycol solution as the stationary phase.[1, 2] The product had bp 75·6°–75·8°/760 mm (determined by micromethod [24]) and n_D^{20} 1·4364. Van der Bij and Kooyman report bp 75·7°, n_D^{20} 1·4355.

It is to be noted that under the experimental conditions, mentioned above, the pyrolysis of hexahydrobenzylacetate yields directly methylene*cyclo*hexane of about 99 per cent purity.[2]

1-*Ethyl*cyclo*pentene*

*cyclo*Pentanone was converted by Grignard reaction into 1-ethyl*cyclo*pentanol, bp 74·5°–75°/20 mm and n_D^{25} 1·4498. McLellan and Edwards [25] report bp 74·5°/20 mm and n_D^{25} 1·4494. 40 ml of the carbinol were dehydrated in the vapour phase over Al_2O_3 at 200° and 4 sec contact time. Under these conditions the olefinic product is formed in approximately 85 per cent yield and contains 77 per cent of 1-ethyl*cyclo*pentene and 23 per cent of ethylene*cyclo*pentane, as determined by gas–liquid chromatography.[1, 2] The 1-ethyl*cyclo*pentene was separated from the higher boiling isomer by repeated distillation through a Piros–Glover spinning band column. The best portion, amounting to 45 per cent of the total 1-ethyl-*cyclo*pentene and containing less than 1 per cent of impurities, was taken as standard material. The product had bp 106·5°/760 mm and n_D^{20} 1·4428. Turner and Garner [5] report bp 106·2°–106·9°/760 mm; Van der Bij and Kooyman [3]: bp 107·3°/760 mm and n_D^{20} 1·4428.

*Ethylidene*cyclo*pentane*

As mentioned above, this compound was obtained in the preparation of 1-ethyl*cyclo*pentene and was separated from it by fractional distillation. After redistillation the product had a purity of more than 99 per cent, bp 112·0°/760 mm and n_D^{20} 1·4490. Van der Bij and Kooyman [3] report bp 112·6°/760 mm and n_D^{20} 1·4490.

2 g of the ethylene*cyclo*pentane were dissolved in 30 ml of pure 2,3-dimethylbutane and ozonized at −20°. The ozonide was decomposed and the liquid product was identified as *cyclo*pentanone by its infra-red spectrum and D.N.P., which after recrystallization from ethanol melted at 145°–146° (literature, 146°).

3-*Ethyl*cyclo*pentene*

*cyclo*Pentene was converted in low yield (15 per cent) into 3-bromo*cyclo*pentene by the action of N-bromosuccinimide, according to Ziegler.[26] The product was treated with ethylmagnesium bromide under the conditions described by Berlande,[27] to yield 3-ethyl*cyclo*pentene. The olefin was purified by distillation through a Piros–Glover spinning band column, and the best portion, amounting to 65 per cent of the total and containing less than 0·3 per cent of impurities, was taken as standard material. The final product had bp 98·0°/760 mm and n_D^{20} 1·4330. Crane *et al* [4] report 98·1°/760 mm and n_D^{20} 1·4321.

4-*Ethyl*cyclo*pentene*

100 g of *p*-hydroxyacetophenone were converted into 4-ethyl*cyclo*hexanol in 67 per cent yield, under the conditions described by Ungnade and McLaren.[28] The product boiled at 191°–192°/760 mm and was ketone-free. Ungnade and McLaren [28] report bp 191°–192°/760 mm. Oxidation of this compound by boiling nitric acid, according to Vogel,[29] gave a yield of 60 per cent of β-ethyladipic acid (bp 176°–178°/0·5 mm). Thermal decomposition [29] of the acid gave 3-ethyl*cyclo*pentanone, bp 165°–167°/760 mm, n_D^{20} 1·4418. The ketone was converted into 3-ethyl*cyclo*-pentanol by hydrogenation in the presence of Raney Nickel (see Lutz and co-workers [30]). The product had a bp of 166°/760 mm, n_D^{20} 1·4455, and its infra-red spectrum showed the presence of a hydroxyl group (band at 3310 cm^{-1}) and the absence of any absorption in the C=O region. 11 g of the 3-ethyl*cyclo*pentanol were converted into the acetate by refluxing with 30 g of acetic anhydride and 8 g of glacial acetic acid for 48 hours. The ester was pyrolysed at 550° and about 12 sec contact time in a Pyrex glass tube as above, and

gave a mixture containing approximately 58 per cent of 3-ethyl*cyclo*pentene and 42 per cent of the 4-ethyl isomer. Small amounts (about 0·5 g) of the pure isomers were isolated from the reaction mixture by gas–liquid chromatography.[1,2] The 4-ethyl*cyclo*pentene, obtained in this way, was 98·5 per cent pure.

Oxidation

The structure of the compound was proved by oxidation to β-ethylglutaric acid. According to von Rudloff,[31] a mildly alkaline $NaIO_4$–$KMnO_4$ solution was used as the oxidant, after a check experiment had shown that pure *cyclo*pentene was converted into glutaric acid in 95 per cent yield by this method. 96 mg (1 millimol) of 4-ethyl*cyclo*pentene and 30 mg of K_2CO_3 were added to 100 ml of aqueous 60 per cent tertiary butanol and shaken for 30 hours with 90 ml of the oxidant (containing 98·3 millimol of $NaIO_4$ and 1·7 millimol of $KMnO_4$/litre). The resulting solution was acidified with dilute sulphuric acid and, after reducing the excess of oxidant, continuously extracted with ether. The extract was evaporated to dryness and the solid residue (145 mg) recrystallized from a small quantity of 2 per cent hydrochloric acid. Mp 72°–73° C. Day and Thorpe [32] report mp 73° C for β-ethylglutaric acid. (Found: C, 52·58; H, 7·41 per cent. Calc for $C_7H_{12}O_4$: C, 52·59; H, 7·55 per cent.)

ACKNOWLEDGMENTS

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A METHOD FOR THE DETERMINATION OF TRACE AMOUNTS OF WATER IN HYDROCARBONS *

By A. F. PYRAH † (Fellow) and R. S. ROBERTON † (Associate Fellow)

INTRODUCTION

WITH the introduction into the petroleum industry of naphtha reforming involving the use of platinum-based catalysts, there has arisen a need for a simple and accurate method for the determination of trace amounts of water in light hydrocarbons. The method would need to be suitable for use in a routine control laboratory. A search of the literature to find such a method was unproductive and suggested that little attention has been given in the past to the determination of water in hydrocarbons in concentrations of about 10 ppm or below. The only method available (unpublished), involving stripping the water from a large sample by dry nitrogen with subsequent collection and determination in ethylene glycol, was found to be both time consuming and unreliable.

The method which is described in this paper involves simple equipment which can be made up in any industrial laboratory, is reasonably fast, and enables water contents within the range 0–100 ppm to be determined on a 20-ml sample of mercaptan-free hydrocarbons, with a repeatability of 2–4 ppm.

EXPERIMENTAL

The usual commercial Karl Fischer titration apparatus utilizing the "dead stop" technique is satisfactory for water contents down to approximately 0·01 per cent. Below this concentration the sensitivity of the instrument is not high enough to detect a slight excess of water or of Karl Fischer reagent, and in addition the effect of atmospheric moisture presents a serious problem.

In the development of a method there were four main objects which had to be achieved.

(1) To increase the sensitivity of the end point detector for use with sample volume of approximately 20 ml.

(2) To exclude atmospheric moisture.

(3) To introduce the sample without risk of contamination from atmospheric air.

(4) To devise a method of introducing the reagent in micro quantities.

The accuracy of the method depends primarily on the sensitivity of the end point detector. A considerable amount of experimental work was carried out on this aspect of the method, and various modifications of the basic detector circuit were tried. The conventional circuit is based on the application of a small potential across two platinum electrodes immersed in the solution. With methanol as solvent and traces of water present no current flows through the solution. At the end point, however, a slight excess of reagent depolarizes the electrodes and current flows, causing a drop in potential across the electrodes. The end point can thereby be detected by noting when an increase in current or a drop in voltage occurs. The voltage drop gives the more sensitive means of detection and, after experimentation with various modifications, a very high degree of sensitivity in very dilute solutions was obtained by the application of a "backing potential" across a sensitive millivolt meter with high resistance. In this way the addition of as little as 0·01 ml of Karl Fischer reagent (equivalent to 0·03 mg of water) could be detected, and the results obtained were in stoichiometric proportion to the amount of water added.

Another development which was required was the design of a titration vessel which could be kept sealed during the titration and which would allow sample and titrant to be added to the flask without risk of contamination from the atmosphere. A suitable vessel has been designed, consisting essentially of a small Pyrex conical flask with two side-arms. The side-arms are fitted with the platinum electrode pair and a specially designed sample dispenser, while the mouth

TABLE I

Correlation tests

Sample	Water added, ppm	Water content, ppm		
		Theoretical	Operator A	Operator B
Naphtha sample A	Nil		12	11
,, ,,	5	17		16
,, ,,	10	22	21	20
,, ,,	15	27	29	28
,, ,,	20	32	33	33
Naphtha sample B	Nil		9	
,, ,,	40	49	47	48
,, ,,	53	62	63	
,, ,,	67	76	72	
Naphtha sample C	Nil		12	
,, ,,	30	42	40	36
,, ,,	40	52	46	46
Naphtha sample D	Nil		17	
,, ,,	50	67	72	72
,, ,,	60	77		82

* MS received 11 February 1959.

† Mobil Oil Co. Ltd, Coryton, Essex.

of the flask is fitted with a rubber diaphragm. A full description of the apparatus is given later. The problem of the addition of reagent was overcome by the use of a micro-syringe pipette fitted with a hypodermic needle. The additional advantage of the needle tip was the fact that this enabled very small increments (less than 0·01 ml) to be added at a time.

When the complete apparatus had been manufactured and assembled, a series of correlation tests was carried out on blends from a suitable plant naphtha of low water content and free from mercaptans and hydrogen sulphide. This naphtha was used rather than a laboratory dried base stock to avoid the difficulties inherent in obtaining and in handling the latter. Carefully dried glassware and connexions were used during sampling. The results of water determinations carried out on the blends are given in Table I.

DISCUSSION

The results obtained on the prepared blends showed good agreement with the calculated figures, and in view of the extreme difficulty of completely excluding atmospheric moisture during blending operations, the agreement obtained is strong evidence for the accuracy and reliability of the method.

The maximum deviation from the theoretical result was 6 ppm and the maximum difference between two operators was 4 ppm. It seems reasonable to assume on the basis of the overall results obtained that the figure for the original naphtha represented its true water content, although it is possible that there is some bias in the method which has not been determined. Additional evidence as to the reliability of the method, not suitable for inclusion in this paper, has been obtained during its use for routine plant control over a period of more than 12 months, when variations in results obtained have agreed with changes in plant conditions affecting water contents.

Because of the type of sample for which this method was devised, no attempt has been made to adapt it for use with stocks containing mercaptans. It could, however, be modified simply for such stocks using the difference technique described by Loveland *et al.**

By utilizing a syringe type of sample dispenser the method has been used successfully for lubricating oil stocks, including bright stocks. The accuracy of the method for these oils has not been determined.

METHOD

Scope

The method is suitable for the determination of water in light hydrocarbon stocks free from mercaptans and hydrogen sulphide. The range covered by the procedure is 0–100 ppm.

Outline of Method

A suitable quantity of "dry" methanol is introduced into the titration vessel and titrated with Karl Fischer reagent to the equivalence point using a syringe type micro-burette. The sample is added by means of a special sample dispenser, and the titration continued to a second equivalence point. The difference between the two titrations represents the amount of reagent absorbed by the sample, and the water content is calculated from this figure and the water equivalent of the reagent.

Apparatus

(*a*) Titration vessel (Fig 1). This consists of a 150-ml Pyrex conical flask with two side-arms. One

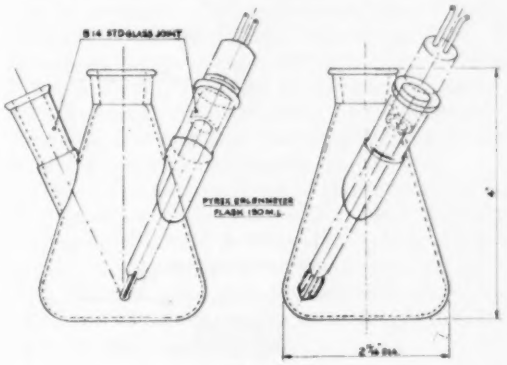

FIG 1
TITRATION VESSEL

arm provides an inlet for a double platinum electrode and is set at an angle such that when the electrode is in position it does not interfere with the action of the magnetic stirrer. The other side-arm provides means of attachment of the sample dispenser. The mouth of the flask is closed by means of a thin rubber cap.

(*b*) Double platinum electrode. This should be of a suitable size to fit the titration vessel. The electrode supplied by Baird & Tatlock (London) Ltd for use on their standard Karl Fischer apparatus is quite suitable.

(*c*) Electrometric detector assembly. A detailed circuit diagram is given in Fig 3. The voltmeter should conform fairly closely to specification, namely,

resistance	100,000 ohms/volt
range	0–100 millivolts
sensitivity	10 millivolts/cm

A suitable type of voltmeter can be obtained from H. Tinsley & Co. Ltd (London). Before use the detector assembly must be standardized according to the procedure outlined in the method.

(*d*) Variable speed magnetic stirrer. The paddle consists of a glass or polyvinyl chloride plastic-covered magnet. The plastic-covered magnet is to be preferred as it is much more robust.

(*e*) Two syringe-type micro-burettes, 0·5 ml capacity, fitted with stainless steel hypodermic

* *Analyt. Chem.*, 1958, **30**, 1316.

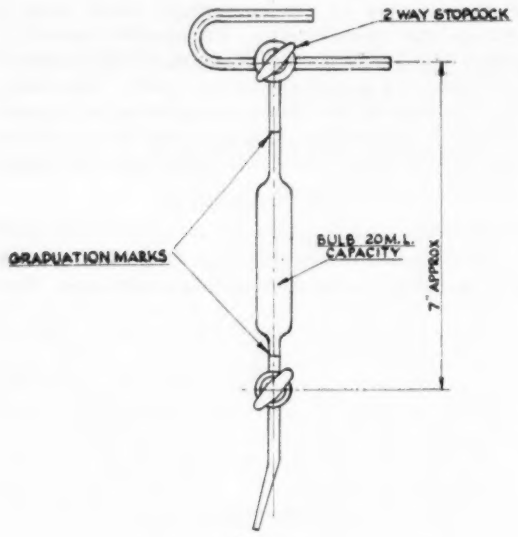

FIG 2
SAMPLE DISPENSER

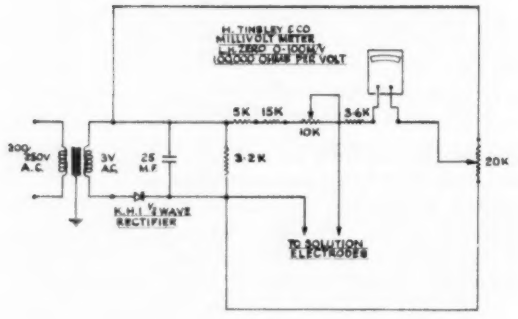

FIG 3
CIRCUIT DIAGRAM

needles (No. 19). These burettes must be capable of delivering and measuring 0·005 ml of solution. A suitable type is the "Agla" micrometer syringe burette manufactured by Burroughs Wellcome & Co. Ltd.

(*f*) Sample dispenser (Fig 2). This is a calibrated glass reservoir of approximately 20 ml capacity fitted with a glass stopcock at each end. The upper stopcock is three-way to permit direct sampling into the dispenser with preliminary line flushing if desired. One end is slightly tapered to fit a rubber bung for insertion into the side-arm of the titration vessel. This rubber bung also contains a hypodermic needle which provides a pressure release during titration.

(*g*) Two 30 ml bottles fitted with rubber diaphragm caps. These are used as reservoirs for Karl Fischer reagent and standard water solution. The micro-burettes are charged from these bottles by inserting the needles through the rubber caps.

(*h*) Sample container. This consists of a 16-oz bottle fitted with a rubber bung and copper inlet and outlet tubes. Attached to these tubes are small lengths of PVC tubing with screw-cocks to enable the bottle to be sealed.

(*i*) Phosphorus pentoxide drying tower.

(*j*) External potentiometer for the accurate measurement of potential.

Reagents

(*a*) Karl Fischer reagent. Water equivalent approximately 3–5 mg H_2O/ml.

(*b*) Standard water in methanol solution 5 mg H_2O/ml.

(*c*) Anhydrous methanol.

(*d*) Phosphorus pentoxide/asbestos mixture for drying tower. Mix 1 volume phosphorus pentoxide with 5 volumes of "Gooch" asbestos.

Preparation and Standardization of Apparatus

The complete assembly is shown in Fig 4. During operation of the equipment the electrode and the sample dispenser must be firmly attached in order to exclude atmospheric moisture.

Place 30 ml of anhydrous methanol in the titration vessel together with a small excess of Karl Fischer reagent. The sample dispenser can be removed temporarily for this purpose and replaced by a B14 stopper. Start the stirrer and add water solution by means of the micro-burette until excess water is

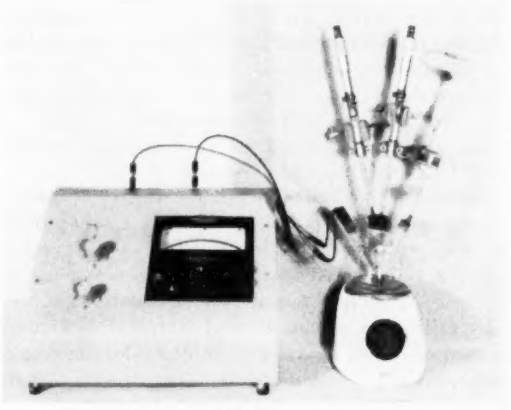

FIG 4

present as indicated by the disappearance of the iodine colour. Add a further 0·1–0·2 ml in excess. This amount is not critical. Operate the stirrer at a speed that ensures efficient mixing without undue turbulence and maintain this constant stirring speed throughout the standardization and subsequent determinations.

The following adjustments must be made with the platinum electrode contacts completely immersed.

Make adjustments as follows using an external potentiometer as indicated.

Connect the potentiometer to the electrode terminals. Depress the release knob on the millivolt meter and adjust the settings of the variable resistances 10 K and 20 K until the millivolt meter reads 100 mV (*i.e.* full-scale deflection), and the potential existing across the electrodes is 400 mV. This adjustment can only be done by trial and error, but by careful manipulation of the two resistances this can be accomplished fairly easily. A small variation of 10–20 mV in the electrode potential setting can be tolerated. However, it is most important to note that the millivolt meter must be in circuit during the adjustments and both readings must be taken simultaneously.

The apparatus is now ready for use. Once set up and standardized no further adjustments should be required.

Procedure

(*a*) *Sampling*

The method of sampling is very important, since serious contamination can result from atmospheric moisture or from improperly dried glassware.

Dry the sample container in an oven and allow to cool in a current of air dried over phosphorus pentoxide. Close the screw-cocks.

Connect the container to the sampling point and allow sufficient naphtha to flow through the bottle to ensure that a representative sample has been obtained. Close the screw-cocks, thus keeping the sample sealed until it is tested.

(*b*) *Method of Test*

Dry all the glassware thoroughly, first in an oven and then by means of a current of dried air.

Connect the sample dispenser to the outlet tube of the sample container and fill the dispenser by gravity flow. This is easily accomplished by inverting the bottle. When full, close both stopcocks on the dispenser and place the dispenser in its position on the apparatus using the rubber bung with the hypodermic needle.

Place 30 ml of anhydrous methanol in the titration vessel with sufficient Karl Fischer reagent (use an ordinary pipette) to " neutralize " the methanol and provide an excess of reagent to absorb any moisture from the air in the vessel. Seal the vessel with the rubber cap, start the stirrer, and allow to stand for a few minutes. Fill the micro-burettes, one with Karl Fischer reagent and the other with standard water solution, by inserting the needle through the rubber cap of the appropriate reagent reservoir bottle and inverting the bottle during the filling operation. Clamp the burettes in position above the titration vessel with the needles penetrating the rubber diaphragm.

Excess of Karl Fischer reagent as shown by the iodine colour should still be present in the methanol, in which case the voltmeter needle will be hard over to the left. Add water solution slowly until the addition of a few drops is sufficient to make the needle travel all the way from left to right. Now add small increments of Karl Fischer reagent at regular intervals (three drops every 3 seconds) until the needle slowly swings back and maintains a position between 0 and 10 mV for a period of 10 seconds. (It is important that the stirrer speed be kept constant.) As soon as the equivalence point is reached observe the reading of the burette. Run in the sample from the dispenser and immediately continue the titration with the Karl Fischer reagent. The second equivalence point is obtained in exactly the same way as the first. It is important to note that the needle must remain steady between 0 and 10 mV for 10 seconds. Observe the second reading on the burette. The difference between the first and second readings gives the volume of reagent used in titrating the sample.

Note: The determination of the equivalence point may present some difficulty at first, since the reaction of reagent with water is fairly slow. After experience in using the apparatus, however, the operator will find that the end point can be identified quite easily. Duplicate titrations on the same sample should not differ by more than 0·01 ml.

(*c*) *Standardization of Karl Fischer Reagent*

This should preferably be done at the time of the test, and in any case the same day. Carry out a determination as described above, but instead of the sample introduce a known amount of water solution. Titrate with Karl Fischer reagent, and from the result calculate the water equivalent of the reagent in mg H_2O/ml. Use this factor in the final calculation.

Calculation

$$\text{Water ppm} = \frac{T \times F \times 1000}{V \times G}$$

where F = water equivalent (mg) of Karl Fischer reagent;
T = titre (ml);
V = volume of sample (ml);
G = gravity of sample.

Precision

Duplicate determinations should not differ by more than the following amounts.

Water present	Repeatability
Below 20 ppm	2 ppm
20–100 ppm	4 ppm

ACKNOWLEDGMENTS

The authors wish to thank the Chairman and directors of the Mobil Oil Company Ltd for permission to publish this paper, which is based on work carried out in the Coryton refinery laboratories. The assistance of F. Bartlett with the instrumentation is also acknowledged.

z

TECHNICAL NOTE

FORMULA FOR CALCULATION OF LEAN MIXTURE KNOCKING CHARACTERISTICS OF AVIATION GASOLINE GRADE 100/130*

By RAOUL NIKLASSON†

THE knocking characteristics of gasolines for piston engines are rated in special laboratory test engines, standardized by the ASTM and IP. These engines are, however, expensive to purchase and operate and it would be valuable to calculate the knocking characteristics on the basis of some chemical and physical data of the gasolines.

Thus, on the basis of real knock ratings in a test engine and some standard analysis data, *i.e.* specific gravity, distillation, aniline point, and TEL content, the following empirical formula for calculation of an approximate value for the lean mixture knocking characteristics of aviation gasoline grade 100/130 is made. The calculated values correspond to the Aviation Method ASTM D 614 and are expressed in performance numbers (and octane numbers for values $\leqslant 100$).

Formula

Knock value = 100 × specific gravity at 20° C + 1/10 × sum of distillation temperatures (° C) at 10 and 50 per cent evaporated + 3/10 × aniline point (° C) − 20 × TEL content (vol per cent)

Test Methods

Specific gravity at 20 °C:	ASTM D 1298
Distillation:	ASTM D 86 (°C)
Aniline point:	ASTM D 611 (° C)
TEL content:	ASTM D 526 (vol per cent)

Examples of calculation according to the formula are shown in the following table:

	Sample 1		Sample 2	
	Analysis data	Formula values	Analysis data	Formula values
1. Specific gravity at 20° C	0·711	+71·1	0·706	+70·6
2. Sum of distillation temperature at 10 and 50% evaporated (° C)	159	+15·9	158	+15·8
3. Aniline point (° C)	54·0	+16·2	60·5	+18·15
4. TEL content (vol per cent)	0·078	− 1·56	0·110	− 2·20
5. Knock value, ASTM D 614	102	—	102	—
6. Knock value, calculated	—	102 (101·64)	—	102 (102·35)

The given formula is applicable to aviation gasolines grade 100/130 according to U.S. specification MIL-F-5572A, British specification D.Eng. R.D. 2485, NATO Symbol F-18, or corresponding specifications.

Analogous formulae might be established for other grades of aviation gasoline.

* MS received 10 April 1959.

† Linköping, Sweden.

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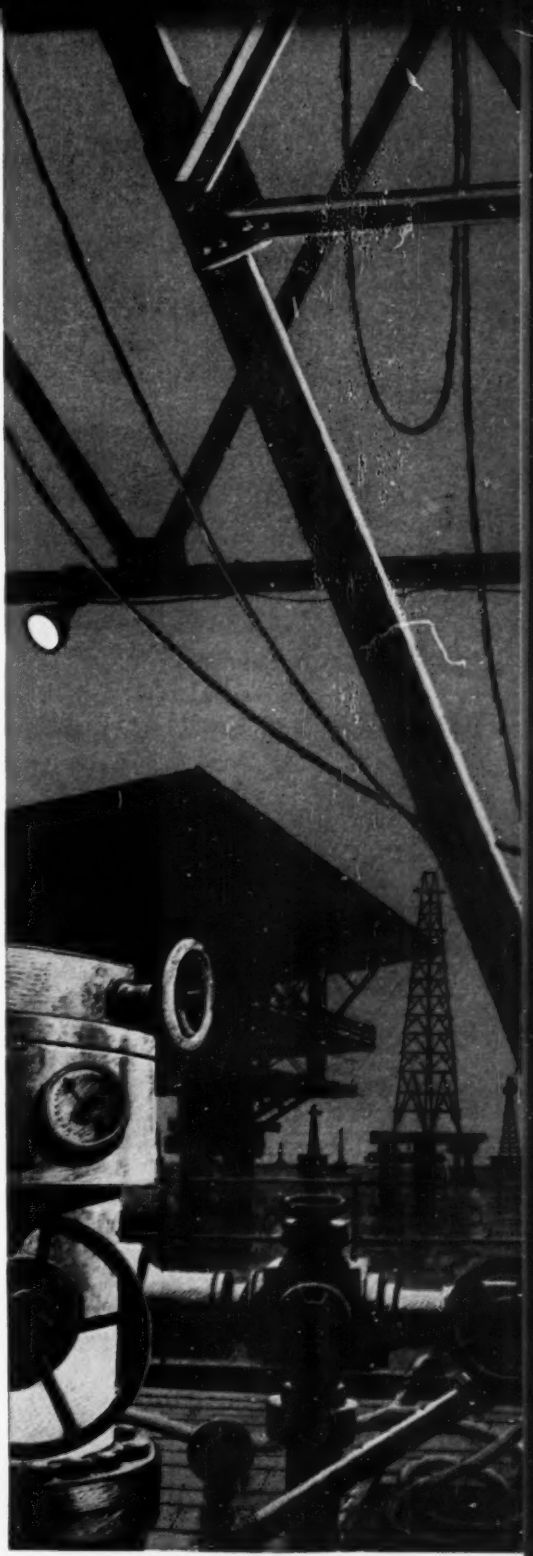

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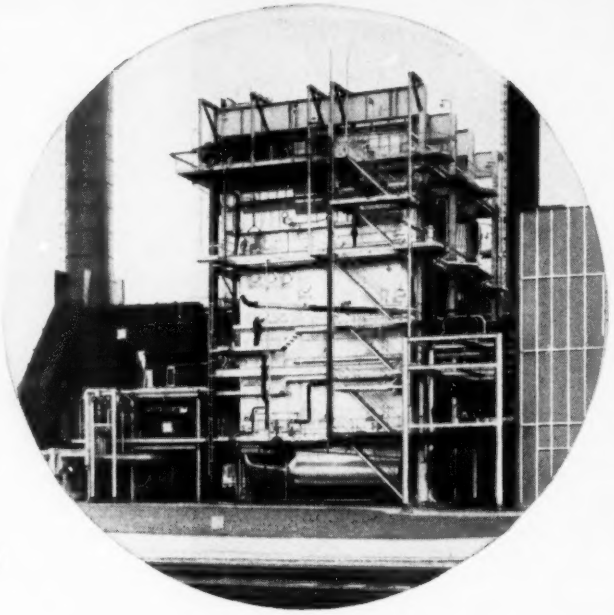

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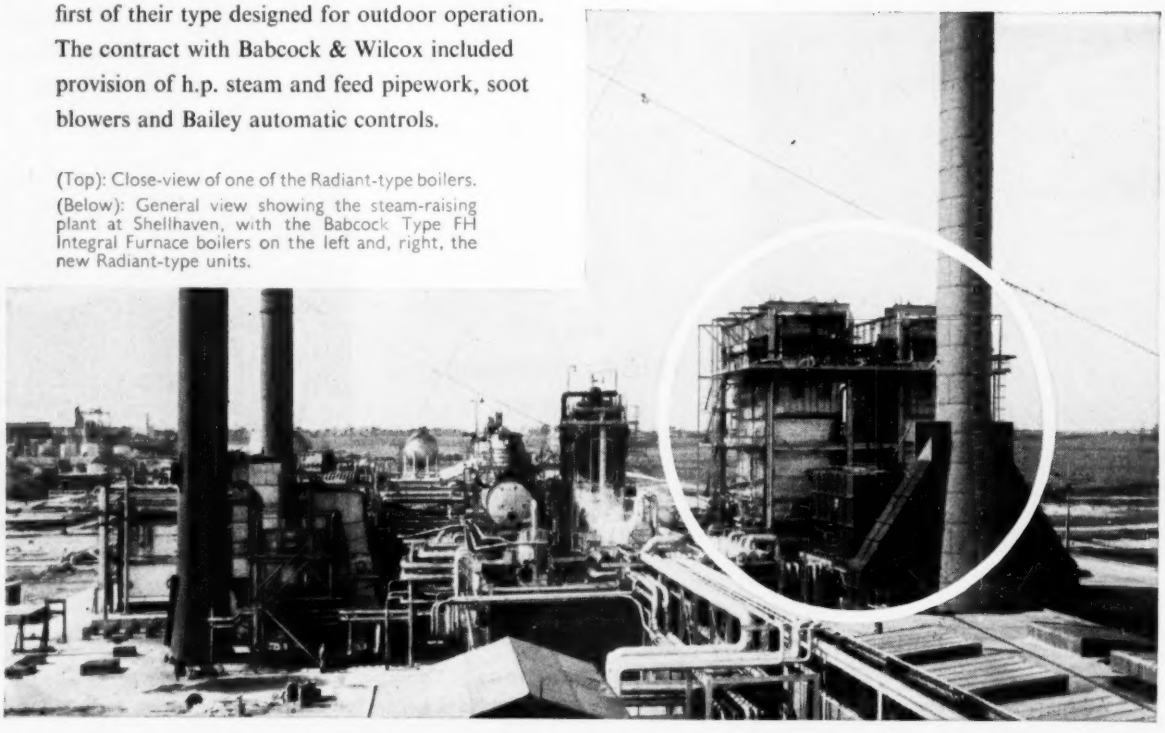
Steam-Raising Plant

(Top): Close-view of one of the Radiant-type boilers.
(Below): General view showing the steam-raising plant at Shellhaven, with the Babcock Type FH Integral Furnace boilers on the left and, right, the new Radiant-type units.



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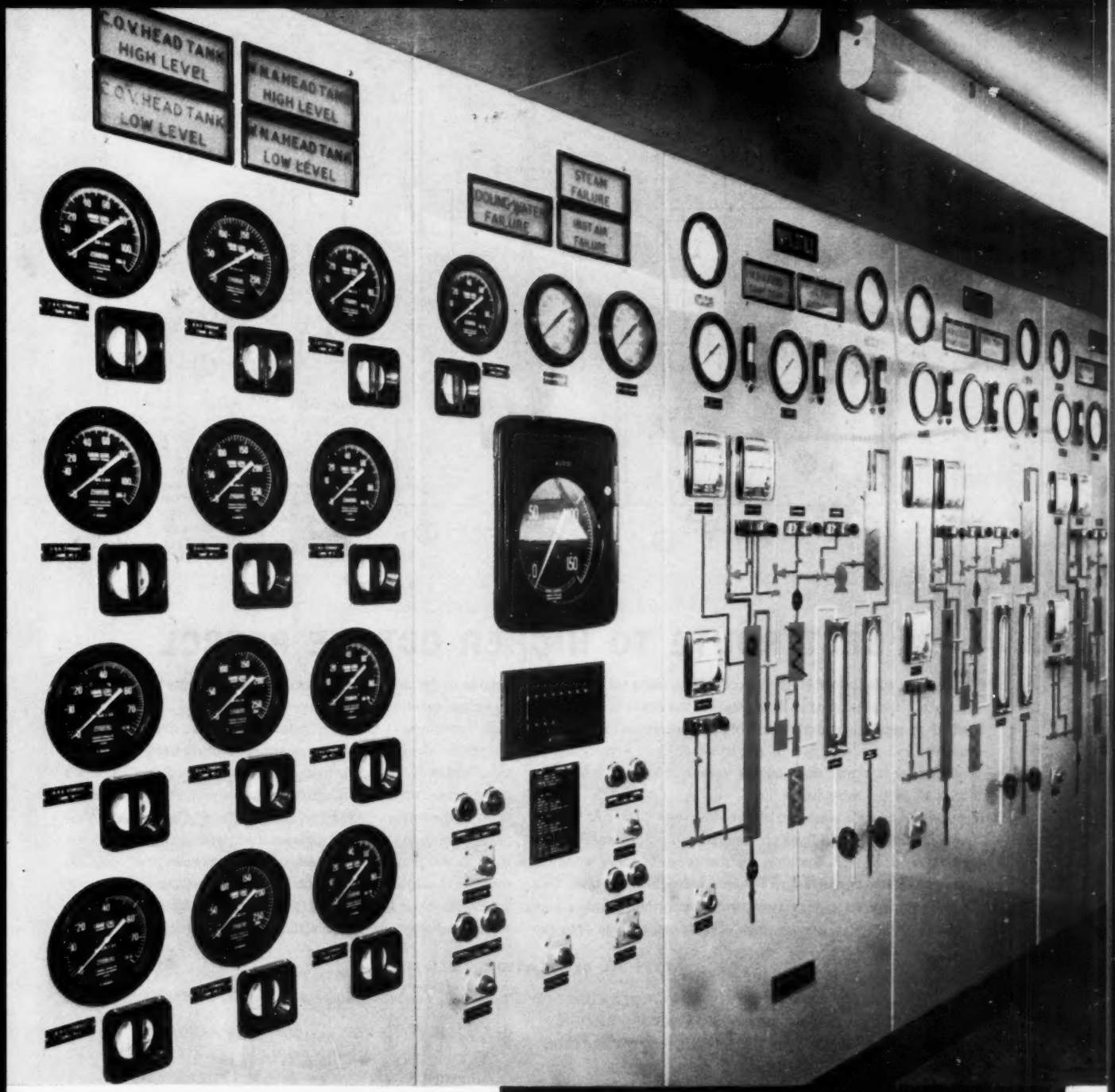

Matthew Hall & Co. Ltd. designed and installed this control panel with associated instrumentation for a chemical plant. It provides a centralized position for the automatic control and indication of temperature, flow, pressure and liquid level, together with a process alarm system.

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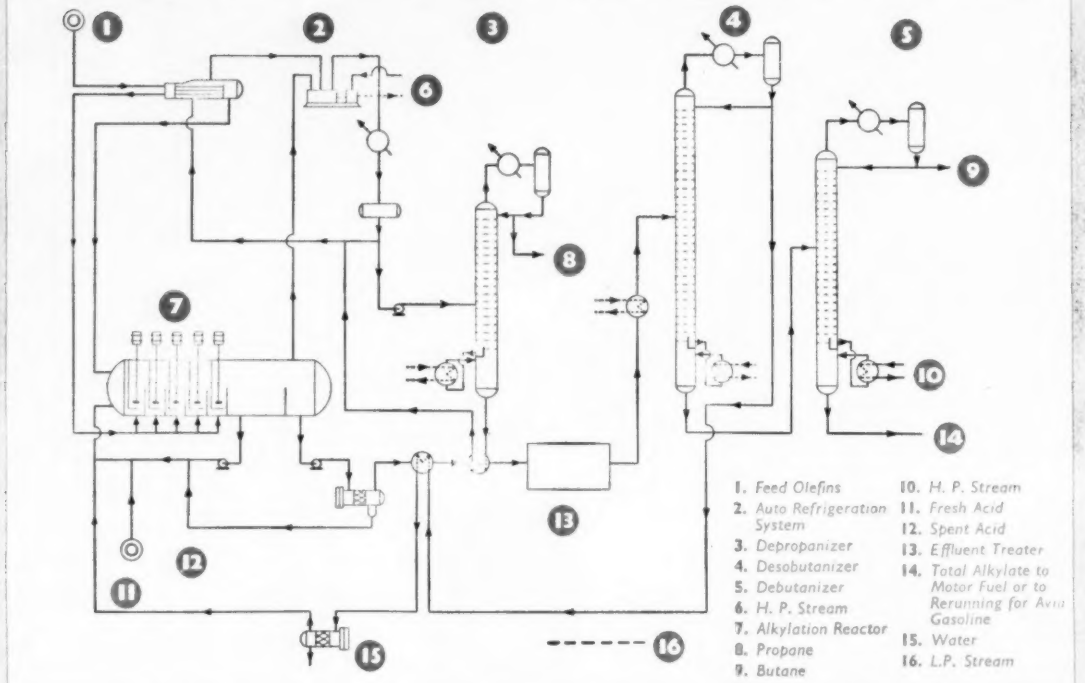

BEST ROUTE TO HIGHER OCTANE PETROL

The Kellogg sulphuric acid alkylation process using the multistage cascade reactor is proving itself throughout the world to be the best method of producing alkylate for high octane petrol. Propylenes, butylenes and pentenes can be alkylated together or individually, producing exceptional high quality material. Typical results are illustrated in the table below.

The Kellogg cascade reactor as shown is a five zone reactor with an internal emulsion pump for each zone and auto-refrigeration. Five streams of olefin feed flow into the five separate zones while the isobutane recycle and acid flow in series through these zones. This arrangement creates a high isobutane concentration and a high isobutane-olefin ratio in each stage, thereby establishing optimum conditions for maximum production of high quality alkylate with a minimum quantity of isobutane recycle.

Acid consumption has been reduced by recent improvements in the reactor design and by utilizing spent acid recirculation plus the use of oleum as part of the fresh acid make-up. Another advantage is as the octane number is increased the acid consumption is decreased. Most of the corrosion problems associated with sulphuric acid alkylation have been eliminated as a result of recent design innovations. Refiners will find this route blazed with the experience of designing over thirty alkylation units, the best way to achieve better quality petrol. Kellogg International Corporation welcomes the opportunity to discuss alkylation with interested refinery engineers.

TYPICAL ALKYLATION YIELD DATA

Olefin Feed Type—	Propylene	40% C_3 60% C_4	Butylenes	Pentenes
Yield—Bbl. Alkylate per Bbl. Olefin	1.78	1.74	1.72	1.60
Isobutane Consumption—Bbl. iC_4 per Bbl. Olefin	1.275	1.174	1.106	0·965
Avg. Acid Consumption: Lbs. 98% H_2SO_4 per Gal. Tot. Alkylate	2.5.0.84	1.5.0.6	0.84.0.33	1.0.0.4
338°F End Point Alkylate Quality				
F.1 Research Octane No., Clear	89.92	92.95	94.97	90.93
F.1 Research Octane No., +3.0cc TEL (WEISE SCALE)	101.9-104	104-107	106.8-109.3	102.6-104.9
F.2 ASTM Octane No., Clear	87.90	90.93	92.94	90.92
F.3 or I.C. Performance No., 4.6cc TEL	117.122	124.127	127.132	116.123
F.4 or 3.C. Performance No., 4.6cc TEL	129.142	141.6.154	150.162	136.148

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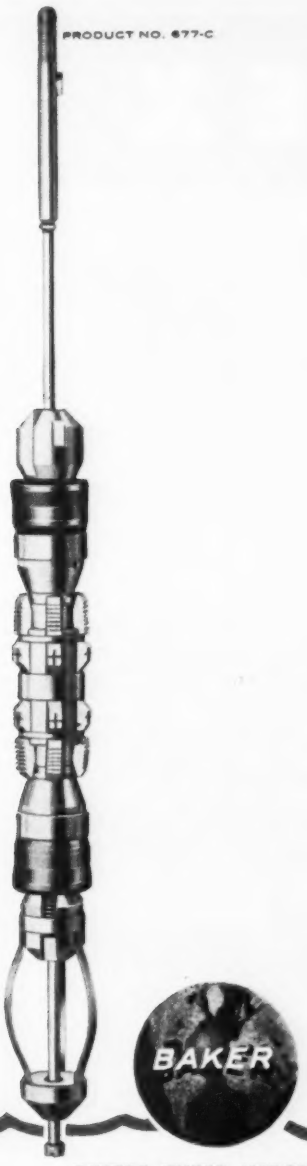

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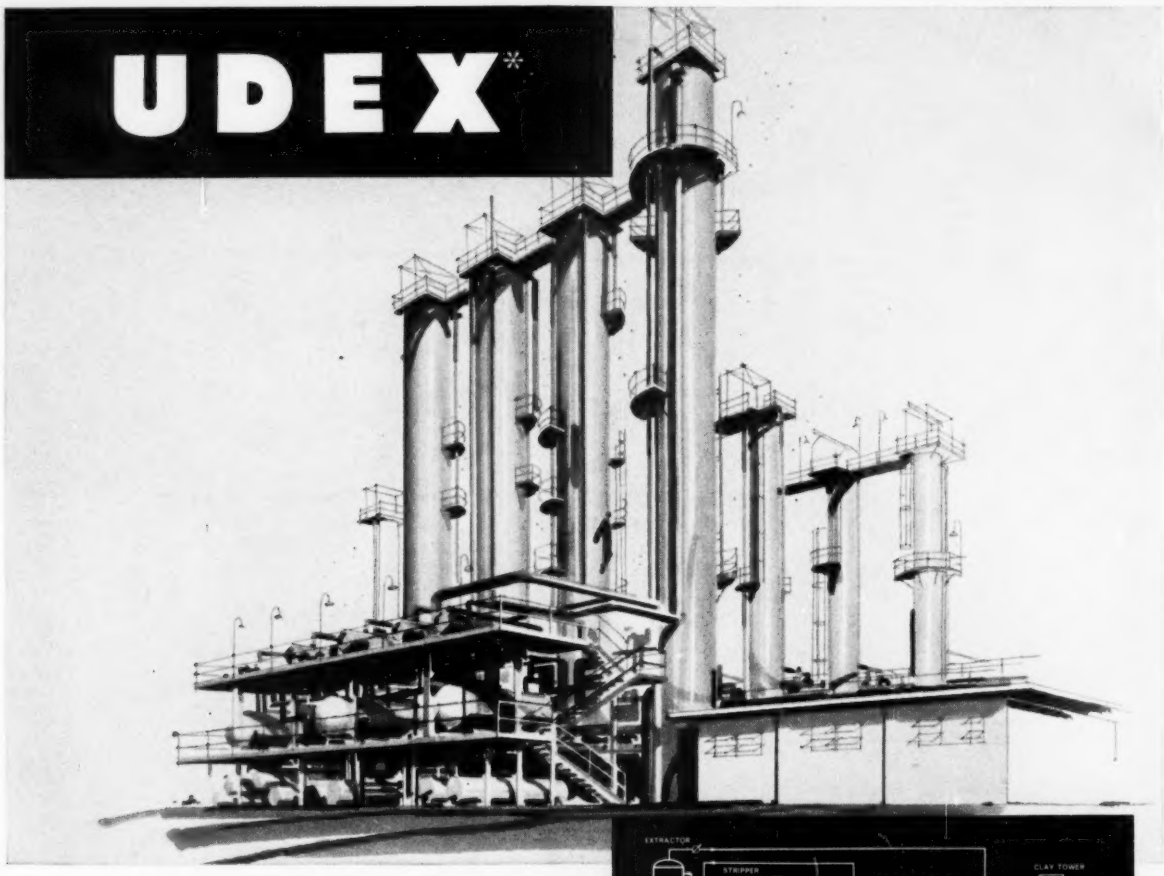

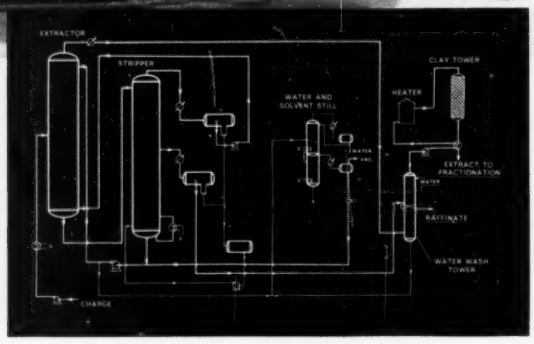

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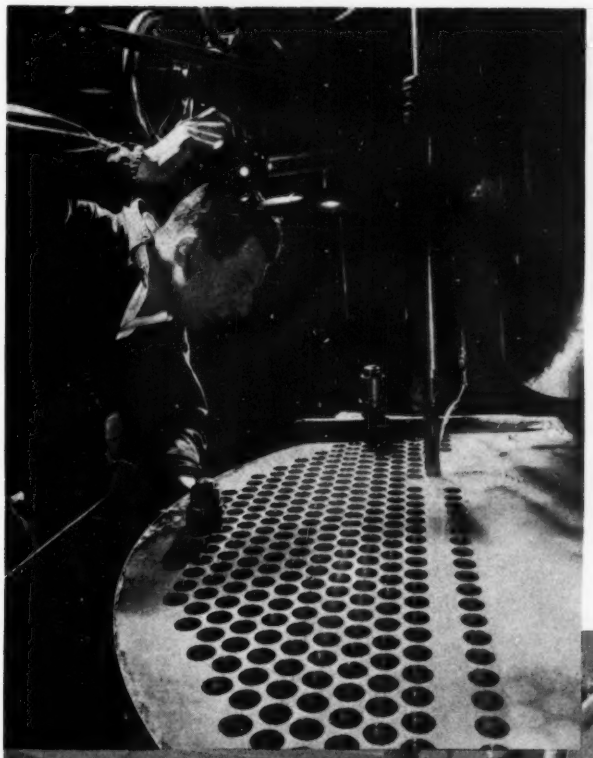

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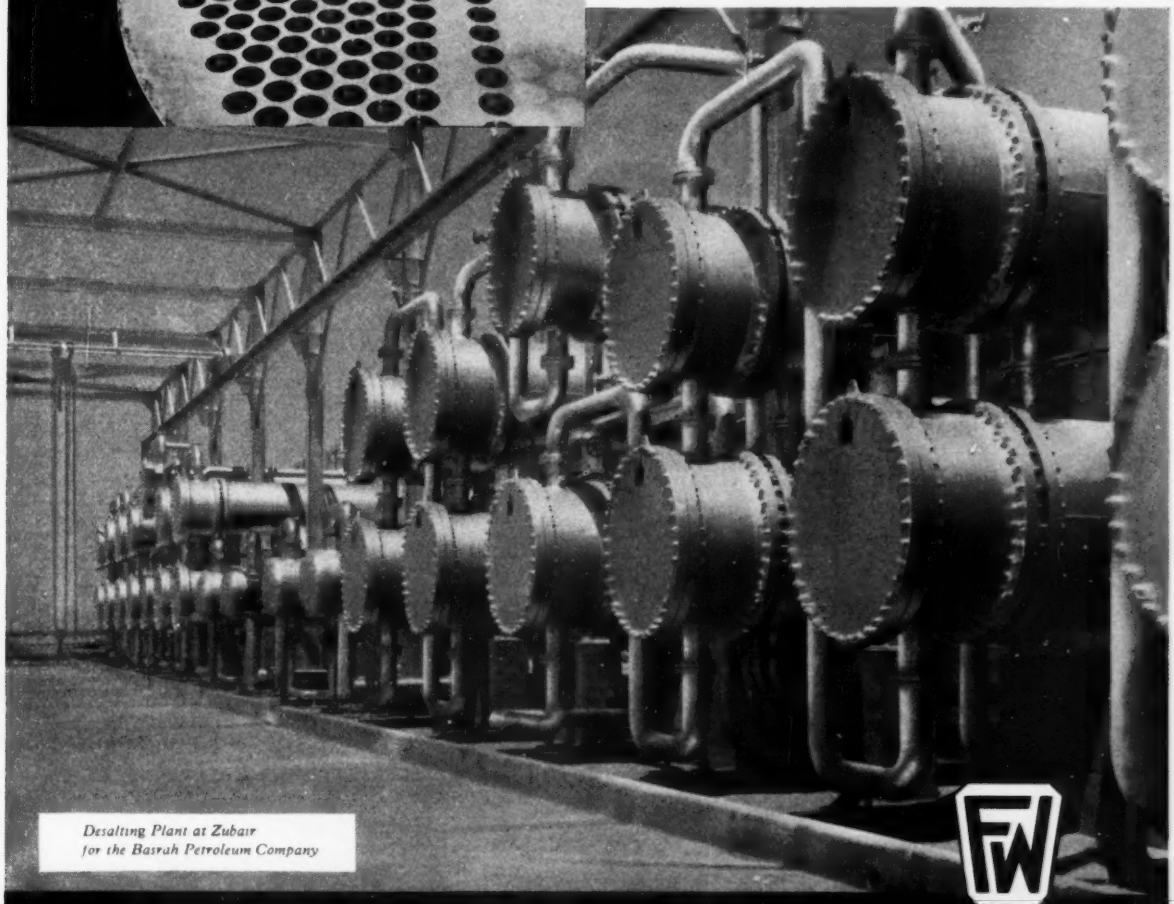

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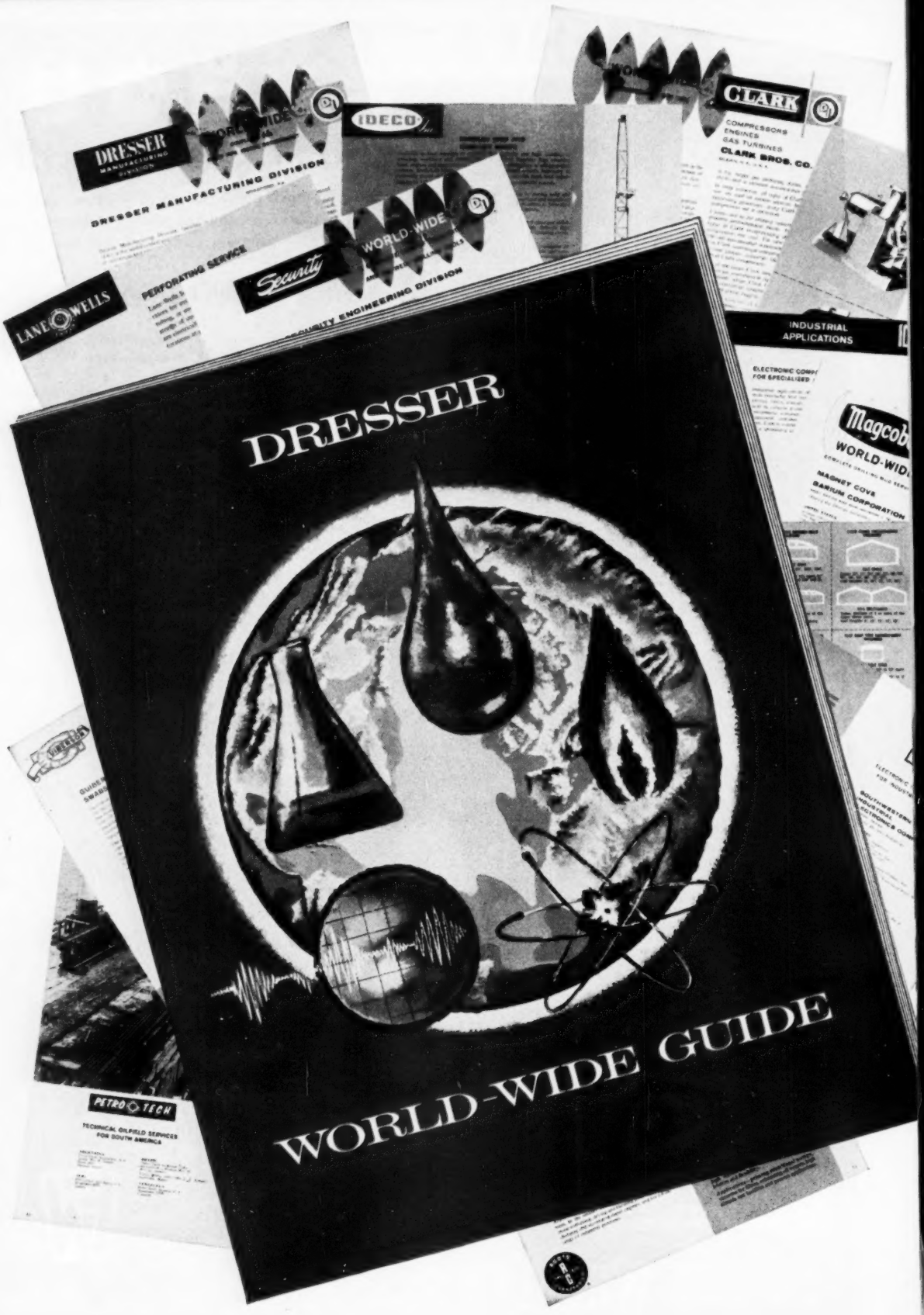
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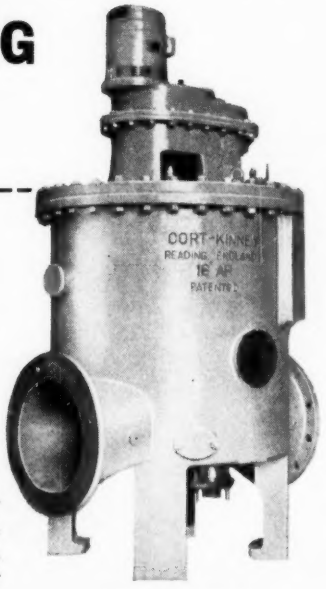

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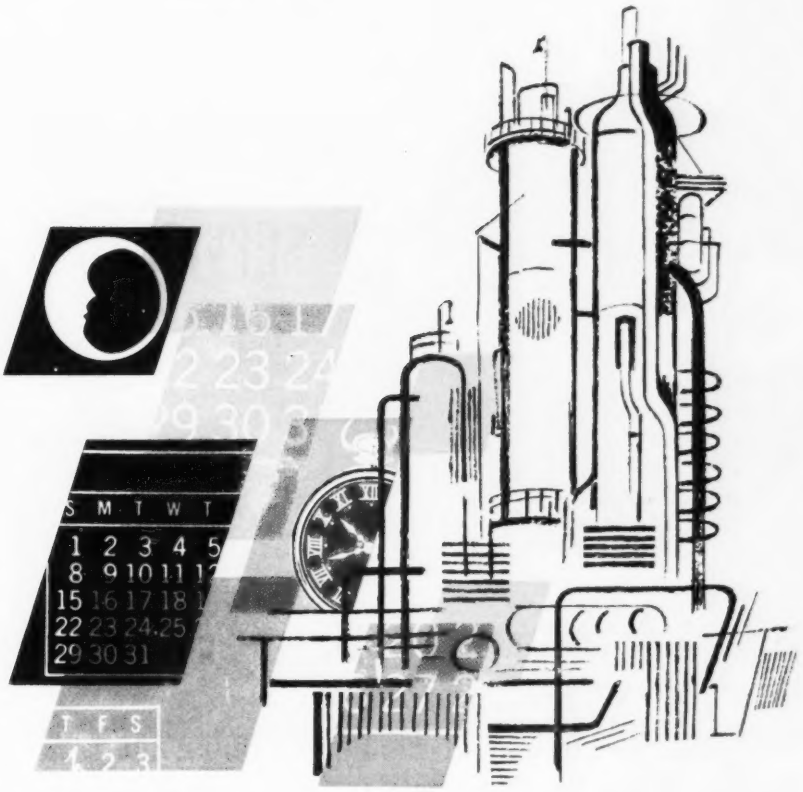

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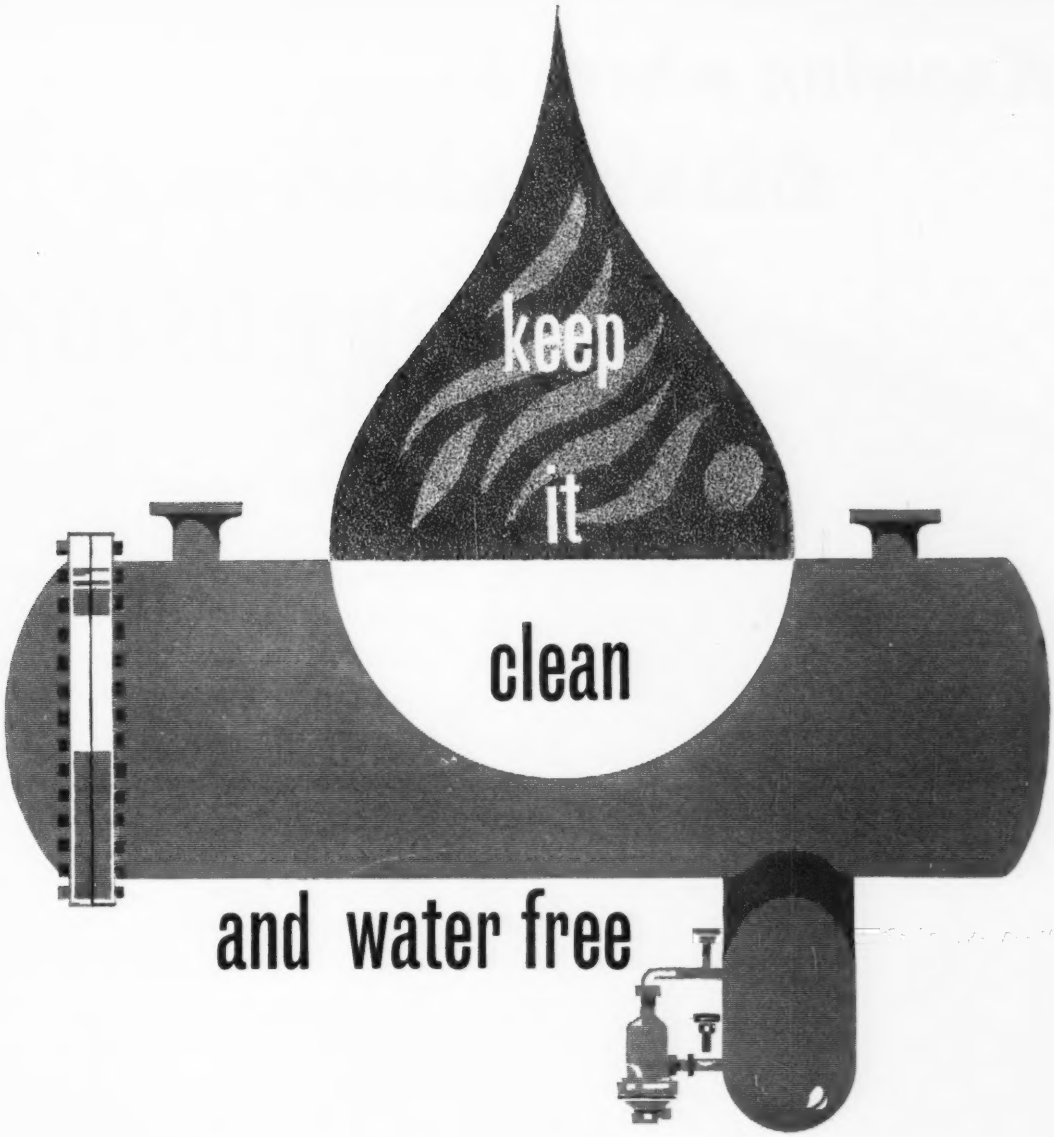

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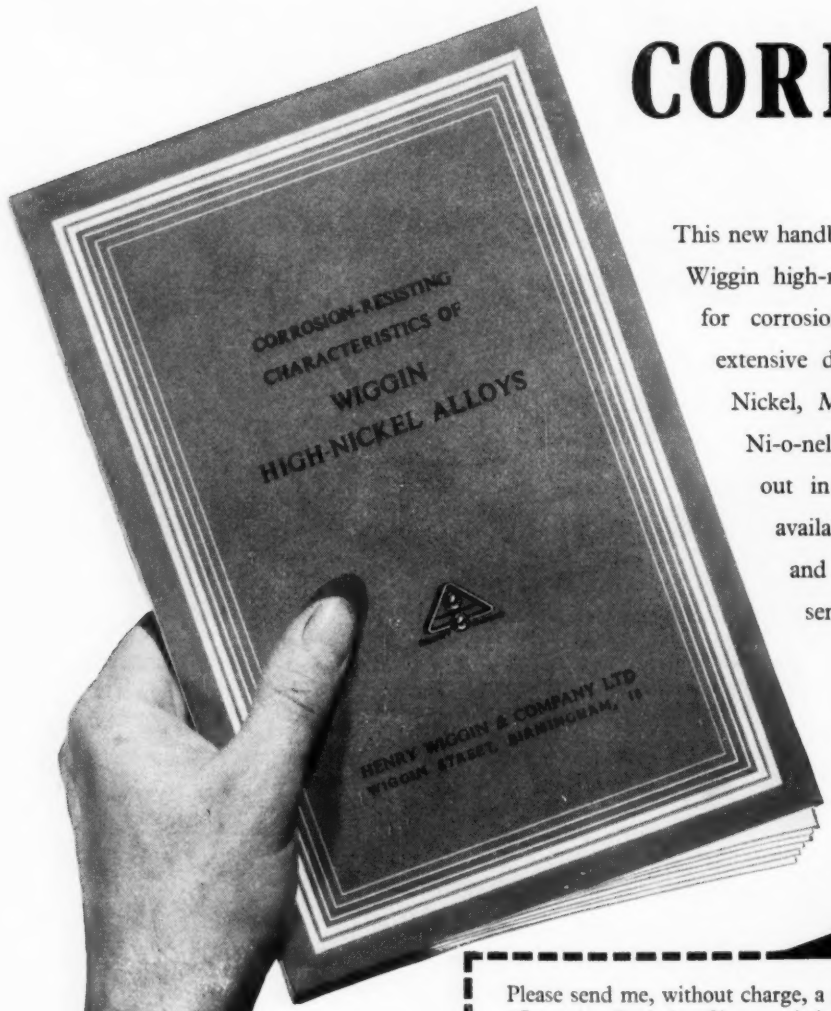

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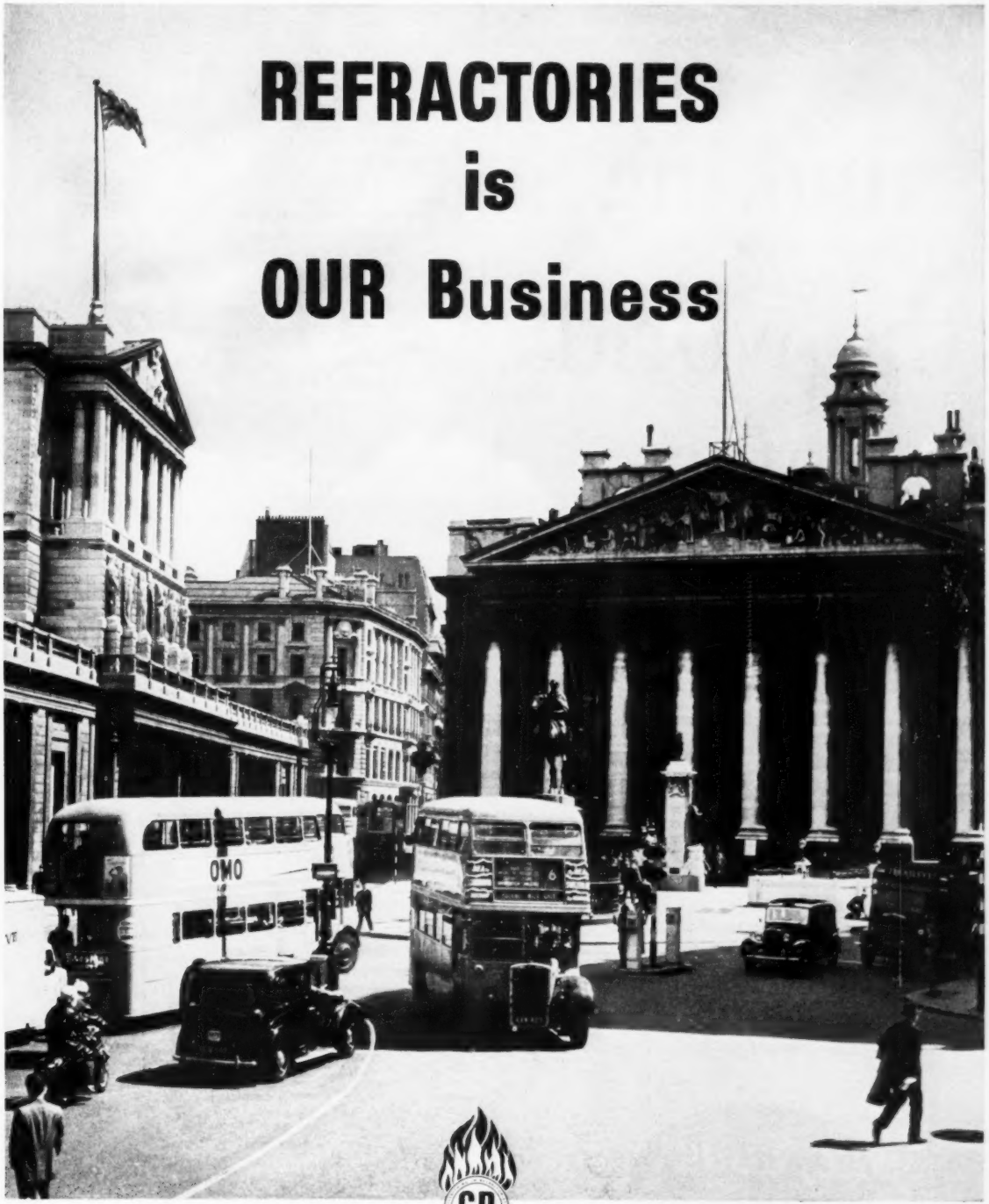
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